Fermilab

October 21, 2005

The attached document describes the upgrade of the SRF Materials Laboratory placed in the Technical Division Industrial Building 3 as a support for the SRF technology at Fermilab.
At this moment the laboratory is used to operate the eddy current scanner which is one of the main steps of the quality control of Niobium, the material used for SRF cavities fabrication.

The upgraded infrastructure will include:

- A cryogenic test station including helium vessel and pumping station which will allow performing RRR, thermal conductivity and Kapitza conductance measurements.

- A fume hood which will allow performing: sample preparation, electropolishing R&D (on small samples) and BCP.

The single subsystems of these two units are described in detail and technical calculations are provided where necessary. Although the required engineering note for the pressure vessel of the cryogenic test station is still being examined by the FNAL technical committee, structural calculations and relief devices design are attached to this document.

The document itself consists of a general section and eleven appendixes. In the general section are introduced all the systems and subcomponents relative to the infrastructure upgrade including: water, ODH, ventilation, hood, safety devices, electrical, and cryogenic system.
In the appendices are reported all the technical specifications and calculations in support of the design choices performed.

DOCUMENTS in SUPPORT of
THE DP-18 REVIEW of the IB3-SRF MATERIALS LAB

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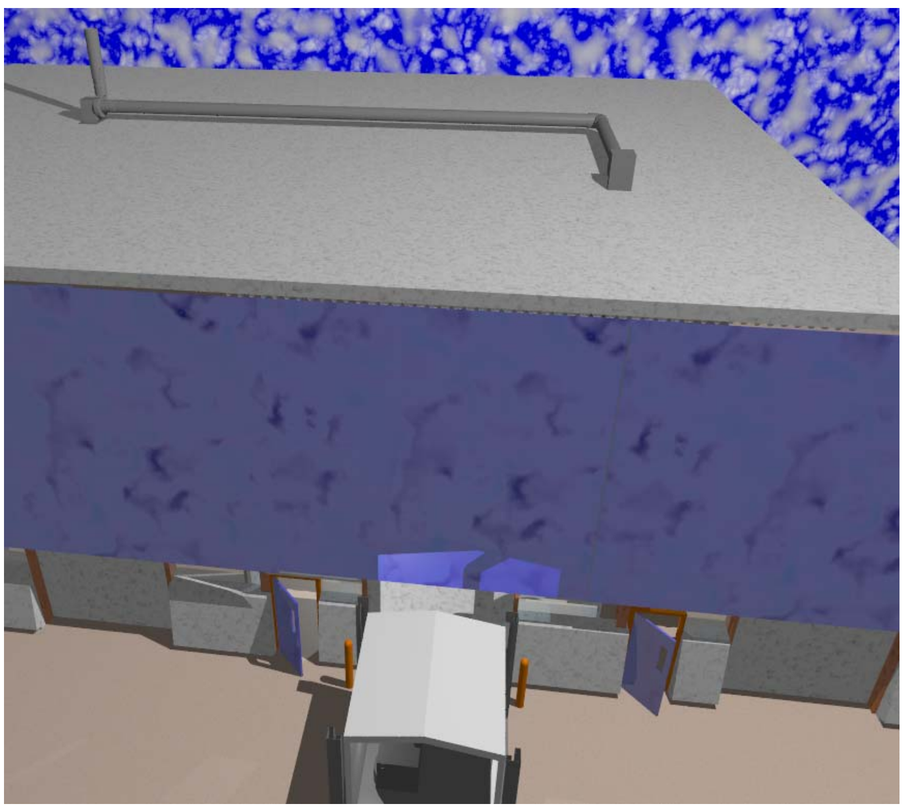

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1) Introduction

Fermilab recently created a new SRF Materials Lab to support the development of SRF technology. Figure 1 shows a schematic of this lab space, including the technical infrastructure that is the subject of this document. The systems we intend to install in the SRF materials lab are:

- RRR Test station;
- Sample preparation infrastructure;
- Sample storage infrastructure;

The RRR measurements are the most important tests performed on the niobium for the cavity fabrication. In the mid-term, however, many other activities should also unfold in this laboratory. Among them are: thermal conductivity, mechanical and magnetization measurements on small samples of the material used in the SRF cavity fabrication. Possibly, these measurements could be performed in the same cryostat as the RRR measurements. For the purpose of this report we will therefore discuss only the RRR measurement system in detail. Further activities, which we plan for the SRF materials lab, are small-scale studies of new sample preparation procedures, such as electro-polishing as well as sample preparation in general. The lab will therefore also include a chemistry hood for sample preparation. Obviously sample storage is also an important aspect of the SRF materials lab infrastructure and the necessary storage space will therefore be provided also.

Appendix 1 shows views of the 19′ by 24′ laboratory space in the refurbished IB3 east gallery, before the installation of the technical components sketched in Figure 1.

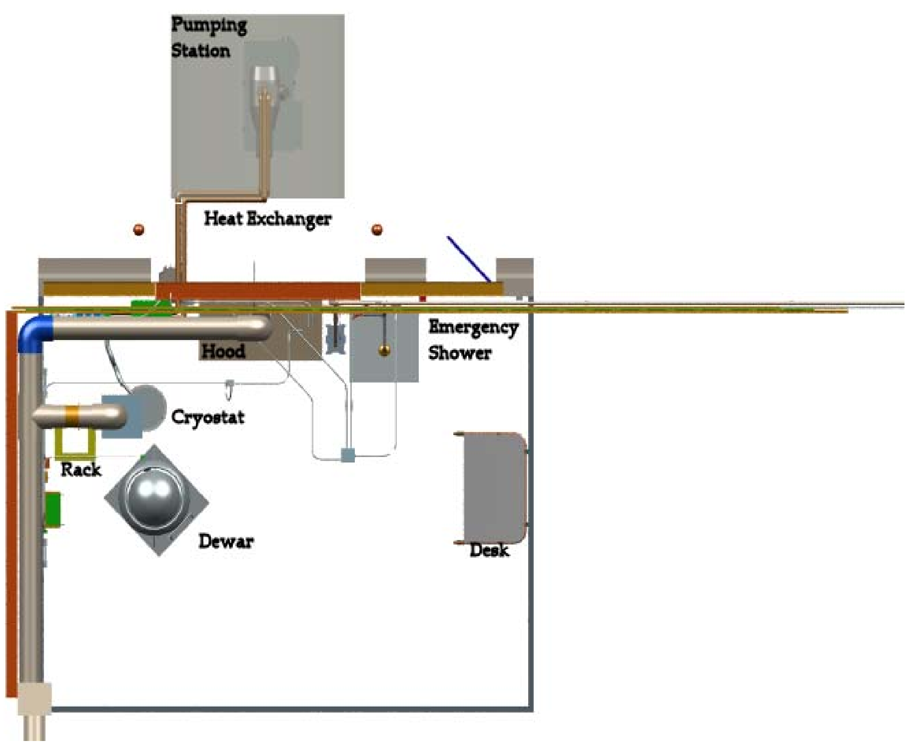


Figure 1: Basic layout of SRF Materials Lab including the RRR test-station and the chemistry hood.

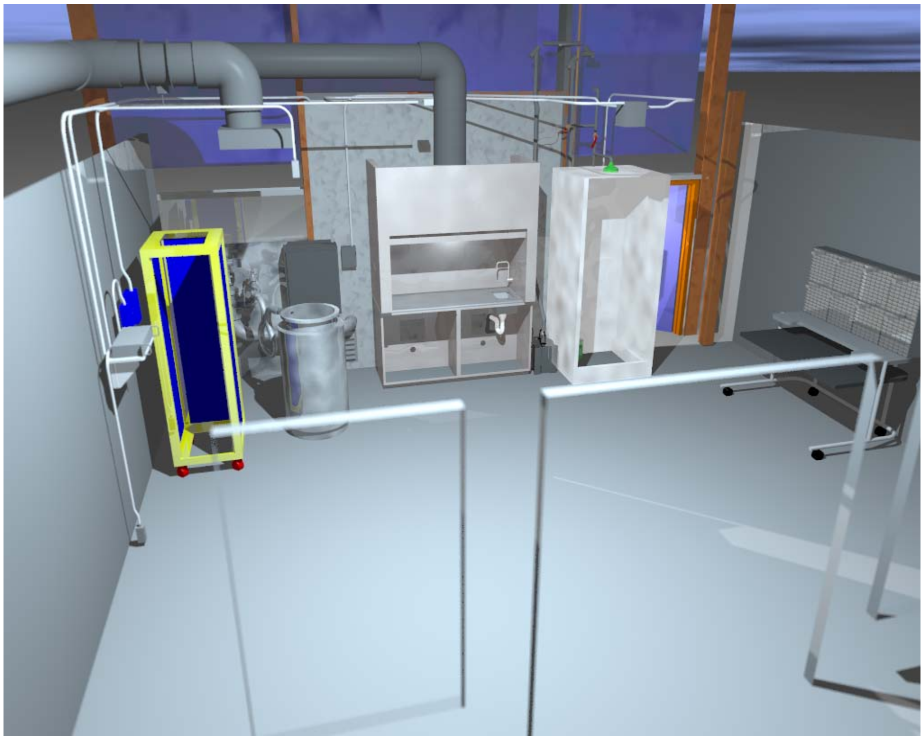


Figure 2: Simulation of the interior view of the SRF Materials Lab.

Figure 2 shows a model of the interior of the SRF materials lab, with all the components discussed in this document. Figure 3 shows an external model-view of the IB3 building showing the shed (transparent in the model) containing the pumps for the RRR test-station.

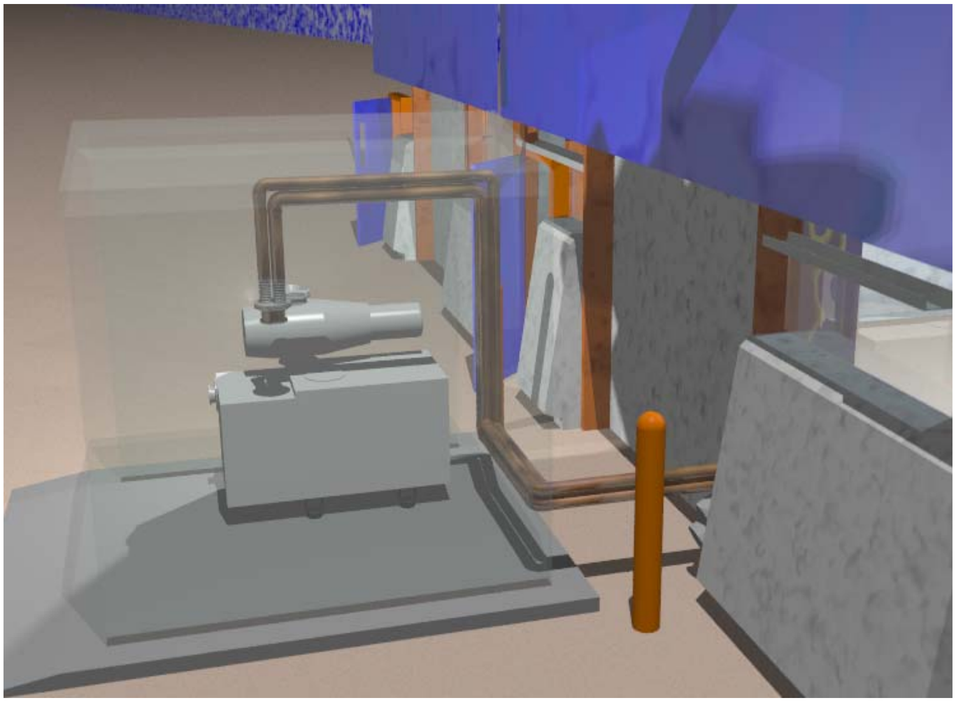


Figure 3: View of the exterior of the SRF Materials Lab.

The following paragraphs will discuss in detail the specific components of the SRF Materials Lab.

2) Chemistry Hood

A recently purchased chemistry hood, shown in the figure below, is to be installed in the SRF Materials Lab as the main infrastructure item needed for the SRF materials sample preparation. The purchased unit includes a blower which will be placed on the IB3 building roof for fumes extraction. The details of fumes extraction and ventilation system are discussed later in this document (2.4). The chemistry hood includes a ventilated acid storage area. The storage cabinets will accommodate containers of up to 1 gallon of acid each, according to the division regulations, which will be needed for the etching and electro-polishing of small samples of SRF materials. These chemical processes typically involve highly corrosive concentrated acids such as: nitric, sulphuric, phosphoric and hydrofluoric.

Chemistry hood for the SRF Materials Lab.

As a preamble to the detailed description of the components and their assembly a general statement needs to be made. With the start of operation of the IB3 extension, projected for 2007, and the ensuing close proximity of the new materials development lab (which will move to the new IB3 extension), the chemistry hood in the IB3 SRF materials lab will become obsolete (since the new MDL will feature several chemistry hoods). There is an agreement (see attached memo in appendix 2) that in this case the hood facility would be dismantled and possibly transferred to the new MDL.

2.1) General Discussion

The following discusses the different major components of the chemistry hood as well as their assembly in the IB3 SRF Materials Lab.

The hood is a 6′ by-pass Labconco Premier Protector Model with a 72×30 in^2 work area. The 1″ thick work surface is made from corrosion resistant epoxy resin suitable for the acid mix we intend to use. The work surface is dished to contain spills. The frame is made from corrosion resistant epoxy coated steel and aluminum. Its design includes sash stops to minimize the operator exposure. The hood also includes a domestic water sink. Figure 4 shows a solid model of the chemistry hood assembly in the IB3 SRF Materials Lab. The hood is placed against the eastern wall of the room. Next to it are an emergency shower/eye wash station and an emergency exit to the east parking lot. The wall behind the hood was formerly a garage door, now covered with a thin, simple, two-layer wall which simplifies the task of providing the needed penetrations. Also clearly visible in this layout is the fumes ductwork which extends from the hood to the area in the NE corner of the laboratory where the Dewar and cryostat for the RRR measurements are installed. From there the ductwork is brought to the NW wall of the lab where it shoots vertically up to the roof of the building where the fan and the chimney are located. This PVC ductwork is embedded in the dropped ceiling. The design of the fume extraction system allows using the chemistry hood blower to prevent ODH (oxygen deficiency hazard) in case of a large-scale liquid helium or nitrogen spill. This secondary use of the blower will be discussed in further detail in a later section of this report. The emergency exit door is equipped with a panic bar.

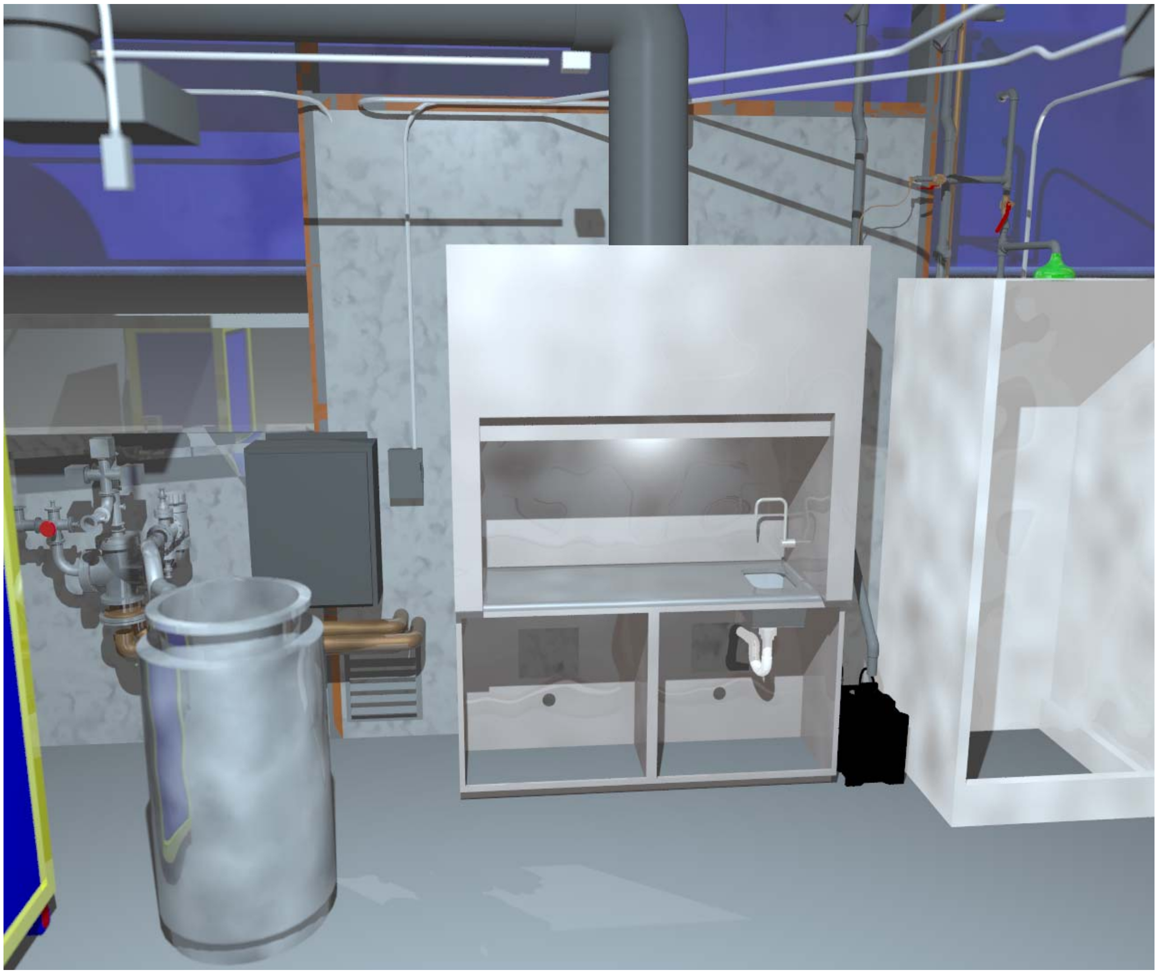


Figure 4: View of chemistry hood assembly in SRF materials lab – view from the inside.

Figure 5 shows a solid model of the view of the SRF materials lab from the outside. This view shows the PVC chimney that guides the chemistry fumes from the hood via the blower to the atmosphere.

The ductwork can be divided in three portions. The first one inside the lab consists of 12″ schedule 40 PVC piping. The second portion consists of an 18′ high rectangular cross section vertical duct that runs along the inside wall of the building. The third portion consists of a ~15′ long 10″ schedule 40 PVC piping running on the roof of the building. The chimney consists of a 10″ diameter, schedule 40 PVC pipe with solvent welded connections at the ends. The chimney is directly mounted on top of the blower and is supported in four points by wiring bolted directly to the roof girders which provide the appropriate electrical grounding. The blower is connected to the rigid duct trough a bellow that adsorbs the motor vibrations. The tip of the chimney is a 4.75′ long "zero-pressure cap", with a multi-concentric tube design that prevents condensation or rainwater from descending into the chimney. A small metallic grid will prevent larger debris or birds from entering into the chimney.

In order to avoid condensation of the fumes during extraction, the speed in the chimney should be 2000 fpm or higher. Detailed discussion and calculations of the fumes speed in the ducts and chimney are reported in appendix 5.

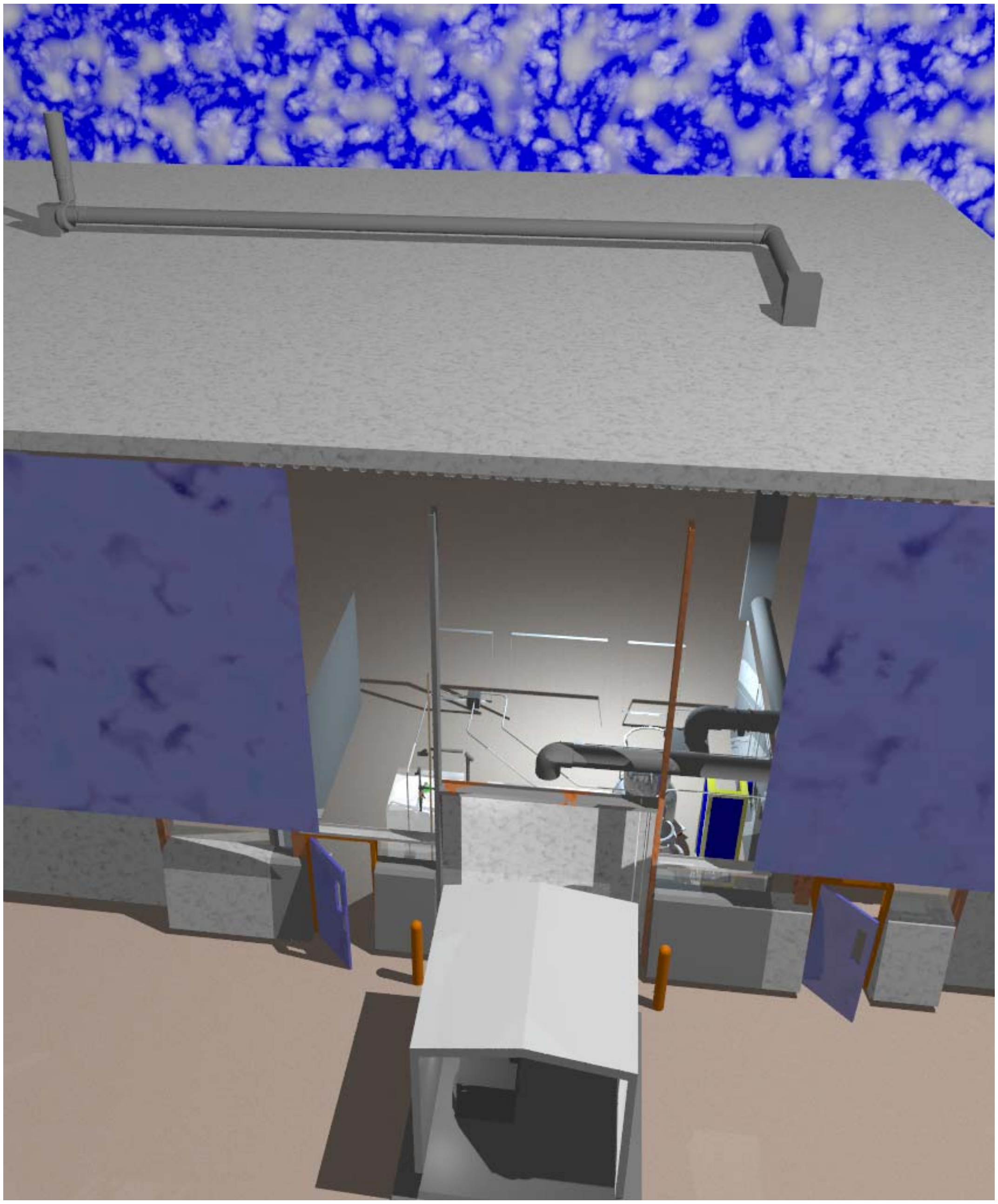


Figure 5: View of chemistry hood assembly in SRF materials lab – view from the outside.

2.2) Blower

The electric blower is part of the Labconco unit package. It is rated 1200 cfm @ 1.1″ wg. The electric power is supplied via a line provided on the roof of the building. Further details on the electric circuits supplying the various components of the Labconco hood are given in another section of this report. The total pressure drop in the ducts, as discussed in appendix 5, is ~1.15″ wg which allows for flow rates above 1100 cfm. The piping on the roof is 10″ in diameter to guarantee sufficient speed of the fumes to leave the chimney and at the same time to avoid condensation in cold weather. The inside portion of the ductwork has a cross section or an equivalent cross section (in the case of rectangular ducts) of 12″ to reduce the losses.

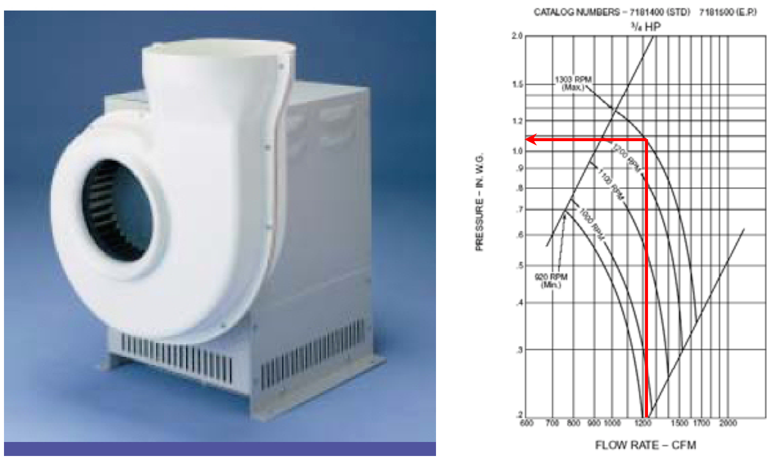


Figure 6: Remote blower for the Labconco Premier hood.

2.3) Safety Shower

The major safety component required in conjunction with the chemistry hood is the safety shower and eye wash station. The safety shower is activated by a pull rod with a D-handle. A solid model of the safety shower assembly is shown in Figure 7. The main components (apart from the shower cabin) are the water supply and drain, the sump-pump, the level sensor and alarm system and eyewash station (Figure 8). Water is supplied from the neighboring Instron lab via a ¾″ PVC pipe running within the dropped ceiling. When triggered, the water from the shower (or eye-wash station – see Figure 8) is collected in a small basin at the bottom of the shower. The sump-pump is activated when the collected water reaches a level of ~2″. A flashing indicator light (mounted on the side of the shower cabin) is triggered when the high level sensor is activated. The alarm is not tied into any lab-wide alarm system. Figure 8 shows a sketch of the sump pump assembly at the bottom of the shower (including the floating level sensor). Since the sump-pump cannot drain the first two inches, an external pump (e.g. a small portable pump unit) will be used to remove the remaining water (e.g. after shower testing). Also shown in the figure is the drain line: the water is pumped back up to the ceiling from where the drainpipe returns the water to the neighboring lab. The return pipe (made of PVC) is slightly tilted at ~-0.6° (1/8″ over 1′) to avoid water stagnation.

According to OSHA regulations, the water supply line is designed to deliver at least 20 gpm (for 15 minutes) to the shower and simultaneously 4 gpm to the eyewash station. Since the typical pressure of the lab domestic water is 40 PSI a PVC pipe with a minimum diameter of ¾″ is needed to keep the pressure drop in the 25′ line below a safe value of 15 PSI.

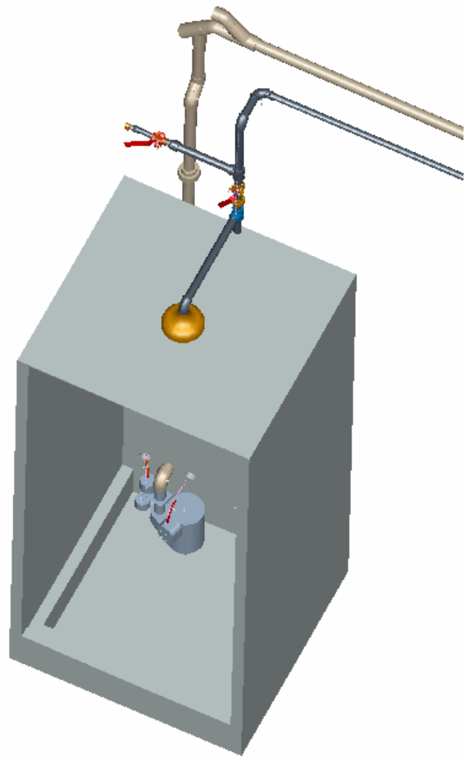


Figure 7: View of chemistry hood assembly in SRF materials lab. Detailed view of the emergency shower assembly.

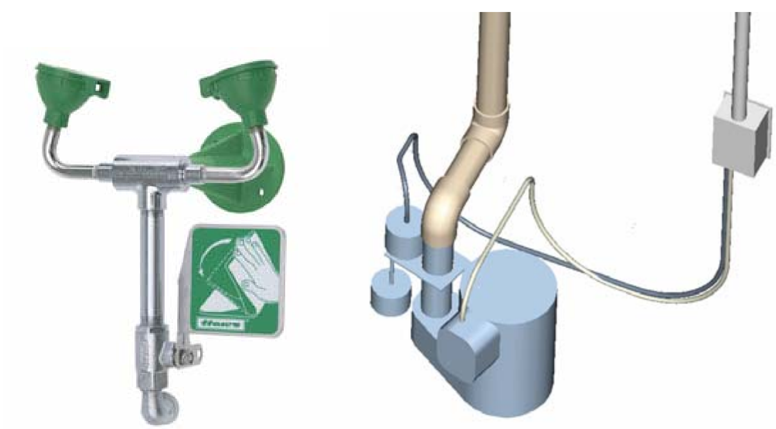


Figure 8: Left: eye-wash unit in Haws emergency shower assembly. Right: sump-pump in Haws emergency shower assembly.

2.4) Fume Circuit

Figure 9 shows the fume circuit in its entirety. The chemistry fumes are collected in the hood and pumped via the blower and through the chimney into the atmosphere. A normally closed secondary leg of the ventilation duct reaches the RRR measurement Dewar area. Since the RRR measurement cryostat and helium supply Dewar can contain up to 600 liters of liquid helium, an electrically actuated damper, triggered into the open position by the ODH sensor (which also automatically starts the blower if needed), connects that part of the line to the exhaust when needed. The damper uses a normally open spring loaded electrical actuator. This feature allows to guarantee the ODH system to work also in power outage conditions. The blower pumping capacity of 1100 CFM @ 1.2" wg is consistent with the minimum pumping speed needed to cope with a worst-case helium spill accident involving the RRR measurement system. If the hood sash is lifted in an ODH situation, pumping occurs in parallel through the duct leading to the Dewar and the hood. The ODH analysis is discussed in another section of this document.

Also included in the fume circuit is the ventilation of the two acid storage cabinets located underneath the hooded work-surface. Two PVC pipes connect the acid storage cabinets with the gap space between the hood back-wall and the baffle within the hood (see Figure 9, right). This gap space is connected to the blower intake and chimney and serves just the purpose of venting the fumes from the stored acids without directing them through the hood area. According to information received from the vendor the draft through the chimney even in the absence of blower activity is sufficient to evacuate these fumes. Figure 9 also shows that the hood sump-pump enclosure is also vented through the hood.

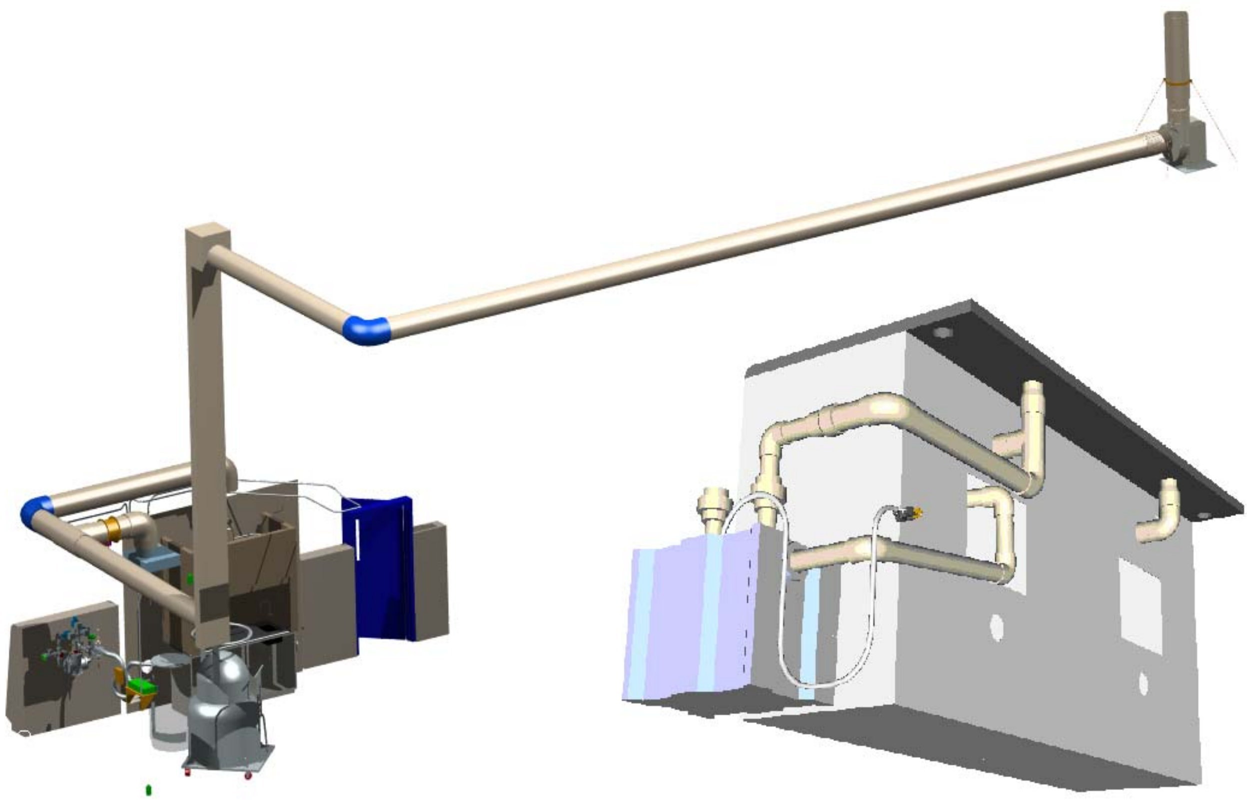


Figure 9: View of chemistry hood assembly in SRF materials lab. Left: complete view of the fume circuit. Right: view of backside of hood assembly.

2.5) Domestic Water Circuit

An important aspect of the installation of the chemistry hood in the SRF Materials Lab is the water circuit. As discussed above, the SRF Materials Lab does not have its own domestic water supply and drain. The emergency shower and the sink installed in the hood need to be supplied from a distribution box in the water system in the neighboring Instron Lab. The return lines drain into the floor drain in the small janitor closet next to the Instron lab (the Instron lab drains there too and precautions need to be taken to prevent the floor drain to be covered or plugged).

The supply and drain pipes are installed in the dropped ceiling. Cleanouts are installed at each elbow to allow cleaning of the pipes without disassembly. The supply lines to the sink can be valved off with a manual valve allowing for maintenance operations. The drain water must be pumped back into the drain line in the ceiling using sump pumps. There are separate sump pumps for the sink and shower. The shower sump pump is shown in Figure 7 and Figure 8. Figure 10 outlines most of the plumbing. The drain lines from the shower and hood sink are shown. Also clearly visible is the sump-pump assembly for the sink. The short stub exiting the closed sump-pump assembly vertically is the first portion of the ventilation pipe, which is also tied into the fume circuit (Figure 9). All drain lines are 5/8” diameter PVC. The water supply lines are copper, 1/4” diameter to the hood-sink, 5/8” inch the others.

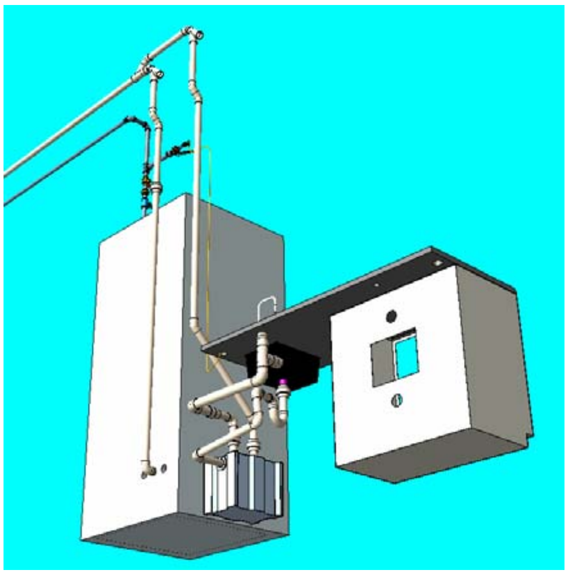


Figure 10: View of chemistry hood assembly in SRF materials lab. Detailed view of the water (domestic) circuit

2.6) Electrical Circuits

The SRF materials Lab will need to address several electrical issues. Present plans call for the installation of a vacuum pumping station with a 10 HP roots blower motor, a 25 HP second stage motor, and a 2.0 kVA control transformer for the motor starters. Due to noise, vibration and heating, the vacuum motors will be placed in a sound shielding enclosure outside the lab, adjacent to the building. The large pumps will be supplied by a special 480 V / 100 A circuit. The pumps are discussed in further detail in section 3.2.

The chemistry hood must have a dedicated circuit for the eye wash station, and separate 110 V circuits for the sump pump and lighting. The work done with the liquid helium Dewar requires a dedicated circuit for ODH monitoring, signaling and damper operation.

The following list details the low power circuits needed in the SRF Materials Lab. The numbering scheme refers to Figure 11.

#	Device	Power	Comment
1	Blower	120 V / 20 A	circuit 1
2	Hood-electrical (lights, plugs)	120 V / 20 A	circuit 1
3	Hood-safety switch	-	manual
4	Shower (level sensor & alarm)	120 V / 20 A GFCI protect.	circuit 2
5	Shower sump-pump	120 V / 20 A GFCI protect.	circuit 2
6	Hood-sink sump-pump	120 V / 20 A	circuit 3
7	ODH-sensors and monitor	120 V / 20 A	reg. power, manual breaker
8	Exhaust butterfly valve	120 V / 20 A	reg. power, manual breaker
9	Vacuum Pumps Controls	120 V / 20 A	reg. power, manual breaker
10	N_2 ODH Fan	120 V / 20 A	reg. power, manual breaker

Figure 11 includes labels corresponding to the items in the above list. The Protector hood is normally wired for 115 V, 60 Hz and 20 A. The junction box (2) for the hood electrical supply is placed on its top surface. The manual hood-safety switch (3) allows disconnecting the hood during repair and maintenance. A similar manual breaker is installed in the circuit supplying the ODH monitor and sensors. Appendix 4 discusses the SRF Materials Lab electrical and ODH issues in further detail.

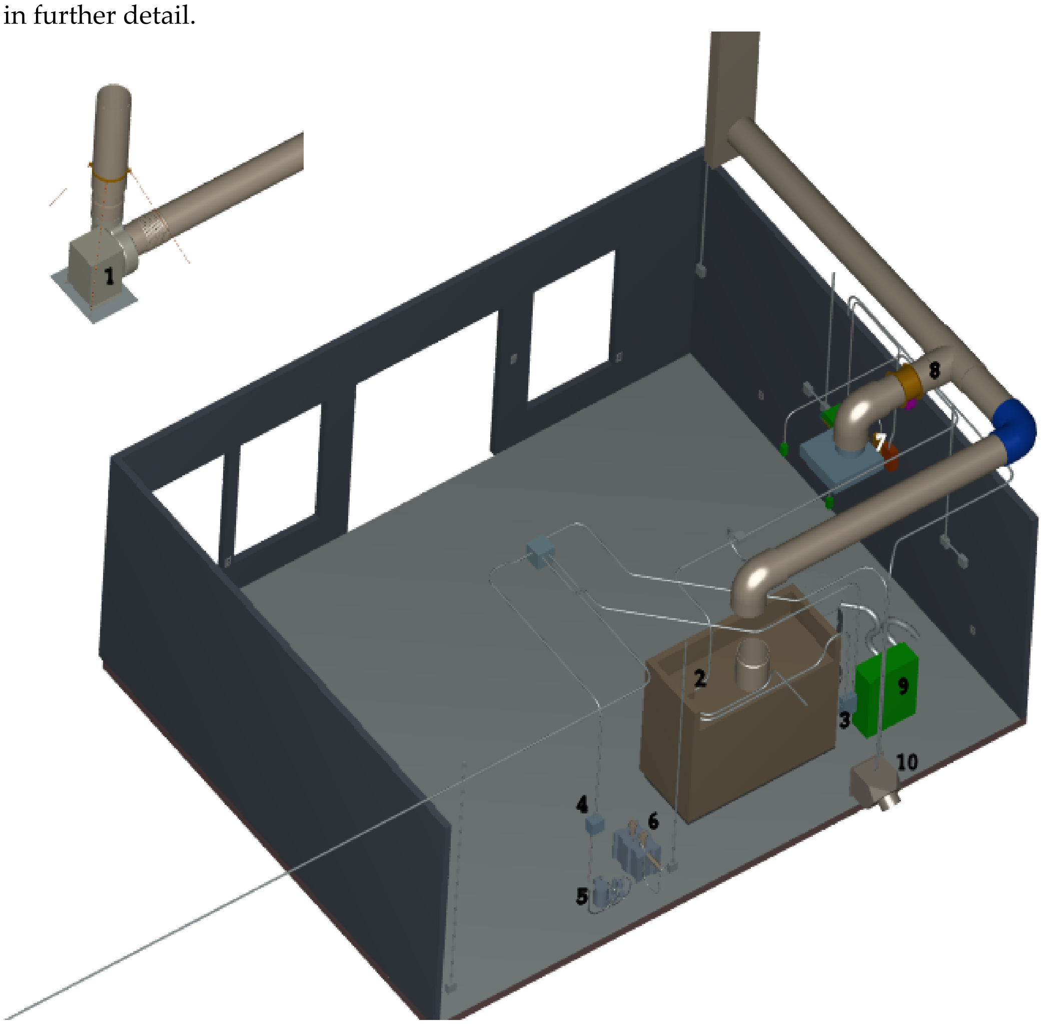


Figure 11: View of chemistry hood assembly in SRF materials lab. Detailed view of the electrical circuits.

2.7) Chemical Safety

The chemistry hood will be needed to perform buffered chemical polishing and electro-polishing of small samples of SRF materials. This typically involves nitric, sulphuric, phosphoric and hydrofluoric acids. The 1:1:2 BCP mix composition, for instance, is: one part of HF (48% wt); one part of HNO_3 (68% wt); two parts of H_3PO_4 (85% wt). The most dangerous chemical for personnel safety in the above mix is the hydrofluoric acid, HF. Further details on the exposure limits as well as possible effects of HF on humans are discussed in the detail safety analysis in appendix 6. Appendix 7 contains the updated (and signed) chemical hygiene plan for the laboratory. All necessary procedures and regulations are discussed there.

Personnel and environmental safety are key issues in the context of the use of the chemistry hood. In the event of a spill or leak involving hydrogen fluoride, persons not wearing protective equipment and clothing should be restricted from contaminated areas until cleanup has been completed. The following steps should be undertaken following a spill or leak:

1. Notify safety personnel.
2. Remove all sources of heat and ignition.
3. Ventilate the area of the spill or leak.
4. If in the liquid form, allow to vaporize and disperse the gas, or cover with appropriate neutralizer (sodium carbonate or an equal mixture of soda ash and slaked lime for HF for example).

The following basic rules need to be followed during routine handling of acids in the SRF Materials Lab:

1. Material hazard sheets need to be attached to all containers holding acid. The labeled containers for the BCP mix need to be made of PVDF-PTFE or HDPE (in secondary container).
2. When handling acids the proper protective clothing (including face shield, lab apron and Tyvek lab coat), gloves and safety goggles need to be worn.
3. The acid should remain as much as possible within the hood area, with the sash closed as much as possible.
4. No acid should be left unattended, not even in the hood, other than in the acid storage cabinet.
5. Sodium-carbonate and sodium bicarbonate need to be in the hood area when using acids
6. Calcium Gluconate should be always in the operation area when HF is involved.

The used acids are to be stored in the acid storage cabinet until they are ready for disposal. The total amount of acid allowed outside of the storage container at any time is to be not more than one gal. The rinsing water needs to be neutralized with sodium-carbonate or sodium-bicarbonate before discharging it through the domestic drain after the pH is checked. The used acid is stored in the proper barrels (see appendix 6) in the storage cabinet for pickup. At any time there should not be more than 1 gallon of stored acid in the acid cabinet. Calcium-gluconate gel (HF antidote) should be readily available for use for immediate treatment of spills on skin.

2.8) ODH

The SRF materials lab could be completely filled with helium gas by a spill from a full 500 liter Dewar. Since helium would rise, the requirement is to have at least as much ventilation from the top of the room as the worst-case spill rate. This was calculated to be 1055 SCFM based on the Circle Seal relief flow rate at full relief pressure in the standard roll-around 500 liter Dewars. Thus, the ventilation rate from the top of the SCRF room should also be at least 1055 SCFM. The chemistry hood blower can provide up to 1100 cfm pumping speed at 1.2" wg and is therefore used as the ventilation component required providing the ODH class 0 standard. The pumping speed provided is assumed sufficient to prevent an ODH situation in the offices above the SRF Materials Lab. In order for the natural infiltration of "make-up" air to occur at a large enough rate a door with an open grid allowing for a 1000 cfm supply is required. Furthermore, the dual use of the chemistry hood ventilation motor and vent controllers requires special labeling such as to warn of the possible ODH hazard starting the fan motor of the chemistry hood when it is disabled. The ODH alarm system over rides local controls.

The SRF materials lab could be completely filled with nitrogen gas by a spill from a full 150 liter low pressure Dewar (and the 35 liters contained in the temperature shield of the RRR cryostat). Since nitrogen gas drops, the requirement is to have as least as much ventilation from the room at floor level as the worst-case spill rate. This was calculated to be 276 cfm based on the rupture disks relief flow rate in the standard low pressure 150 liter LN Dewars. Thus, the nitrogen ventilation rate from SCRF room should also be at least 276 cfm. It is foreseen to install a fan at floor level, next to the hood, that can provide 500 cfm flow rate. This suffices as ventilation to provide the ODH class 0 standard.

In concert with the ODH safety analysis in appendix 3, there will be a sensor mounted high in the room for helium leaks and another mounted low for nitrogen leaks. Both sensors are connected to an ODH control chassis with horn annunciation and strobe lights. Venting of the area will use a damper mounted in series with the fume venting piping. Operation of the damper will be controlled by the motor control card of the ODH unit and an actuator, with positive spring control to open position in the event of a loss of power. The ODH unit also automatically activates the hood blower, bypassing the regular blower switch (which might be in the off position). The nitrogen venting louver will be activated by the louver control card. Note that either sensor triggers both systems ventilation systems in any case.

Appendix 3 discusses the detailed calculation performed in the context of the ODH analysis. Figure 12 shows the components of the ODH protection system, including the hood for venting helium (including blower and damper), the louver with fan for venting nitrogen, the ODH monitor and sensors.

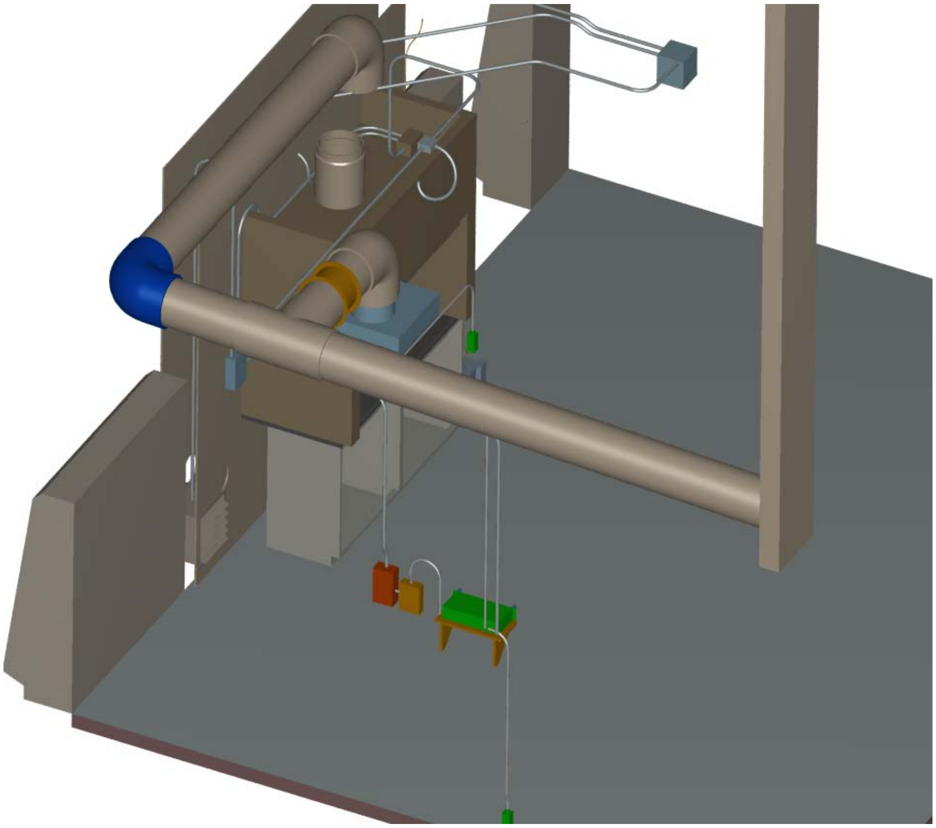


Figure 12: ODH components for SRF Materials Lab.

3) RRR Test-station & Pumps

The RRR (residual resistivity ratio) is a measure of the material purity, for which a minimum threshold is usually specified in SRF applications. The RRR is the ratio of electrical resistivity between room temperature and ~4.2 K. Therefore the RRR test-station allows cool down of the sample to ~4.2 K and measurement of the electrical resistivity.

The low temperature is provided by flooding the volume containing the samples with liquid helium transferred from a transportable Dewar. The intermediate temperatures are set in the sample volume by warming up and streaming helium gas using heaters.

Furthermore the RRR measurement test-station was designed such as to allow for thermal conductivity measurements in the future. For this particular purpose the system needs to provide a helium bath with a minimum temperature of ~1.4 K.

The following discusses the features of the proposed RRR test station that are relevant to this review. This includes a general introduction into the test system design, a discussion of the pumps and a discussion of the cryostat safety issues.

3.1) General Discussion

Figure 13 shows a model of the multi-sample holder for RRR measurements. The sample-holder consists of a central G10 cylinder with copper brackets that allow clamping of the samples as well as current flow, such as to provide all samples with the

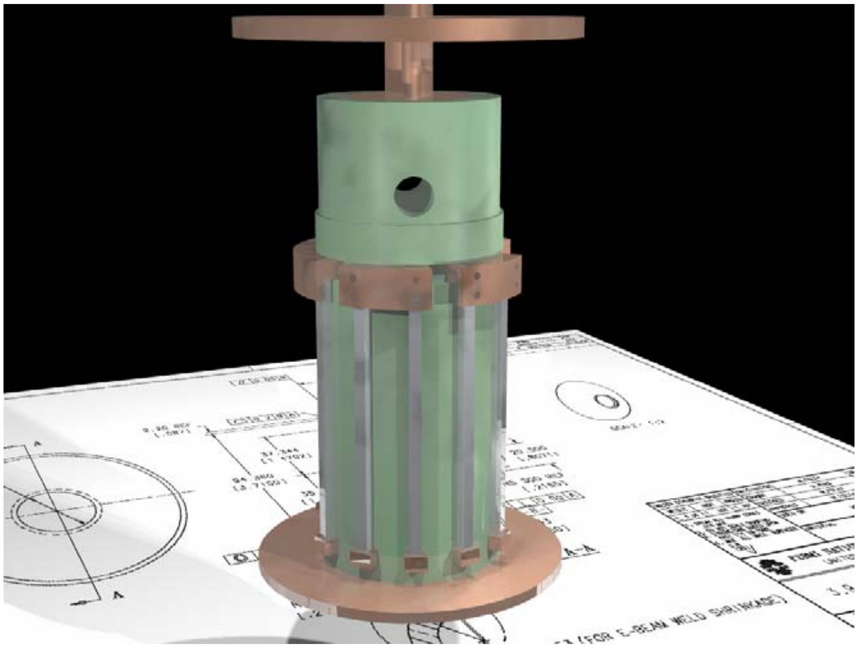


Figure 13: Samples holder for multi-sample RRR measurement.

measurement current. The samples are ~3" long sticks cut from the niobium sheets. The measurement current is sent through all samples in series. The 40 voltage taps needed to measure the electrical resistance use spring loaded battery contacts embedded into the sample-holder on the backside of the samples. The experimental hardware for the RRR measurement system consists of two low power current sources, a precision digital voltmeter (DVM) and a multiplexer that allows switching the signals from the different samples to the precision DVM.

The main aspect of the RRR measurement system of interest here is the temperature regulation. Fig. 14 shows a schematic with the proposed temperature regulation system design. The cryostat acts like a reservoir that contains liquid helium at the boiling point (4.2 K). The liquid helium is transferred by activating the vacuum pumping system connected to the measurement setup vessel via a cold needle valve into a heating chamber embedded in the G10 sample holder. The heating chamber is equipped with four independent heaters that warm up (and evaporate) the liquid helium. The heat exchanger like design ensures that the gas is uniformly heated before reaching the samples. Feedback loops using the readings from temperature sensors placed in the gas-flow allow for automatic temperature regulation. The samples vessel is separated from the cryostat through a vacuum shield, such that its temperature does not affect the bath. This vacuum shield is continuously pumped and also includes multi-layer super-insulation.

The warmed up gas flows through the sample volume regulating the samples temperature. The pumping speed is regulated (see pumping manifold in Figure 15) such as to achieve a continuous gas flow from the heating chamber through the sample for a given helium supply valve opening and heater power. The inside of the sample chamber was designed to minimize thermal mass such as to reduce the thermal time constant of the system.

RRR Measurement Setup

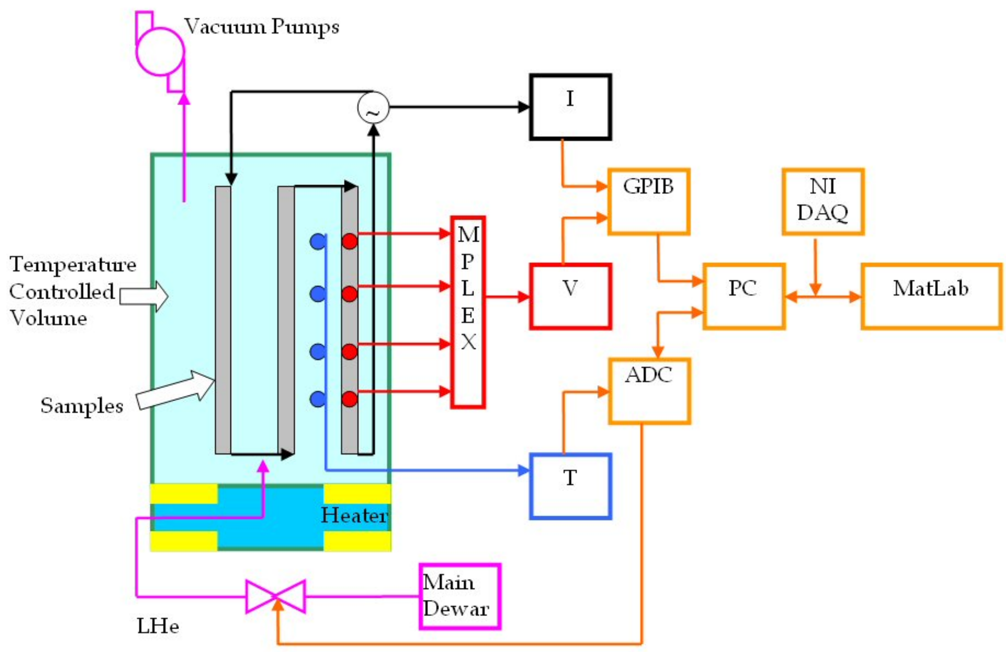


Figure 14: RRR Measurement Schematic.

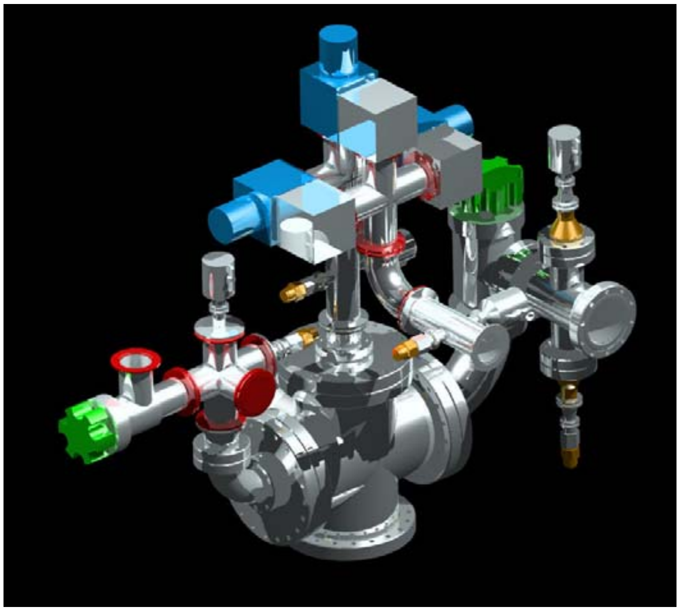


Figure 15: Pumping speed regulation manifold for the RRR measurement.

3.2) Pumping System

The pumping system needed for the RRR measurement system was designed to permit cooling of the complete volume of liquid helium contained in the measurement cryostat to a minimum temperature of ~1.8 K. In this case a pumping system that can pump ~1.4 g/s of helium is needed to ensure a reasonably short pump-down time (1-2 hrs). This is much more than needed to pump much smaller quantities of warmed gas through the RRR measurement system. Therefore the pumps specification was based on the task of cooling down 100 lit of liquid helium to 1.8 K. Appendix 9 discusses the calculations on which the specifications used for the purchasing of the pumps are based. These calculations indicate that the needed combined pumping speed is 1155 cfm at 16 mbar and 300K. The system that is used to reduce the bath vapor pressure to the mbar level in fact requires two pumps – one for the low-pressure end (the root pump) and one for the high pressure end (the backing pump). Typically such pumping systems include a bypass of the root pump for its protection in the high-pressure regime. Figure 16 shows a picture of the purchased Leybold pumping unit. It consists of a 25 HP SV630 backing pump and a 10 HP WAU2001 roots blower. The combined pumping speed is 1140 cfm at 0.1 mbar and 300 K, with an ultimate pressure limit of less than 8 mTorr.

Figure 16: Root (top) and backing (bottom) pumps as purchased from Leybold.

Fig. 17 shows a layout of the pumping circuit, with the pumps (including power panel), the flow regulation system, the heat exchanger and the cryostat. The pumps have to be installed outside of the lab because of noise and vibration issues. Since the location of the pumps is not consistent with future plans for the extension of the IB3 lab, an arrangement was made according to which the pumps will be moved further away as soon as the IB3 extension becomes a reality. This arrangement is detailed in a memo that is included in appendix 8. The pumps will be placed on an elevated, custom made concrete pad and contained in a commercial shed. The pump housing needs temperature regulation including heaters (during the winter) and ventilation (fan and exhaust shutter).

The pumping speed regulation system is also shown in Figure 15. It mainly consists of a manifold, with two branches that can be remotely opened or closed and one branch with a (manual) fine regulation valve. The pumps are provided with 408 V, 100 A / 3-phase power (more on the powering issue in appendix 4).

The heat exchanger is required to ensure that the helium gas is at room temperature before entering the pumps. Calculations and finite element models have shown that the most economic and compact solution consists of three parallel 3" copper pipes seven meters long. Keeping the heat exchanger in the pump system housing avoids frosting in cold weather which could reduce the heat exchange efficiency. The heat exchanger design and calculations are discussed in appendix 10.

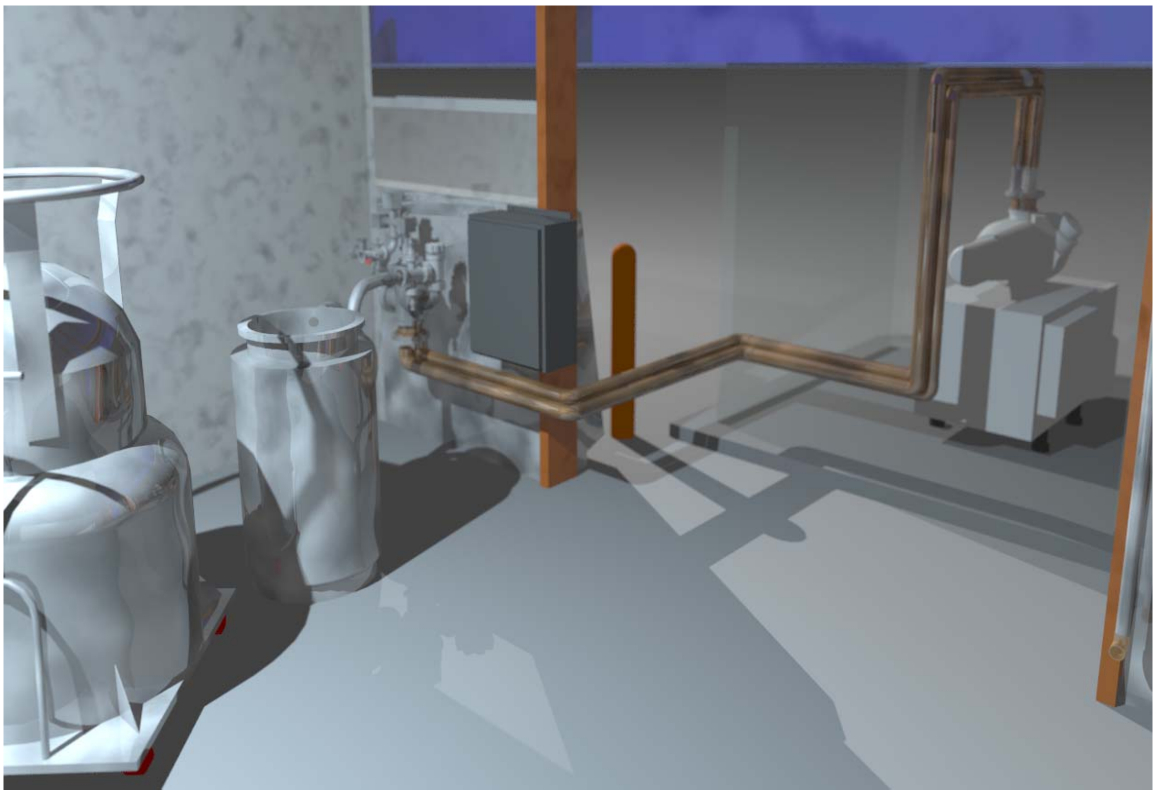


Figure 17: Simulation of the pumping system for the SRF Materials Lab RRR test station.

3.3) Cryostat Design

The cryostat, which will be used to contain the liquid helium and the RRR measurement system, is kept at atmospheric pressure by a custom designed parallel plates relief valve. This feature, avoids the classification of the inner helium container as a pressure vessel. Figure 18 shows a cross-section of the cryostat, as purchased from Precision Cryogenics Inc., including the liquid nitrogen (LN) shield and the vacuum shield. The inner vessel is designed to contain 100 lit of liquid helium. The ODH implications are discussed in section 2.8 of this document as well as in the ODH analysis in appendix 3. The nitrogen shield is considered a pressure vessel and therefore a technical note per FESHM chapter 5031 as reported (DRAFT) in appendix 11 is required.

The vessel is not ASME code stamped, but was made in accordance with ASME code. The respective safety analysis was performed and is given in appendix 11.

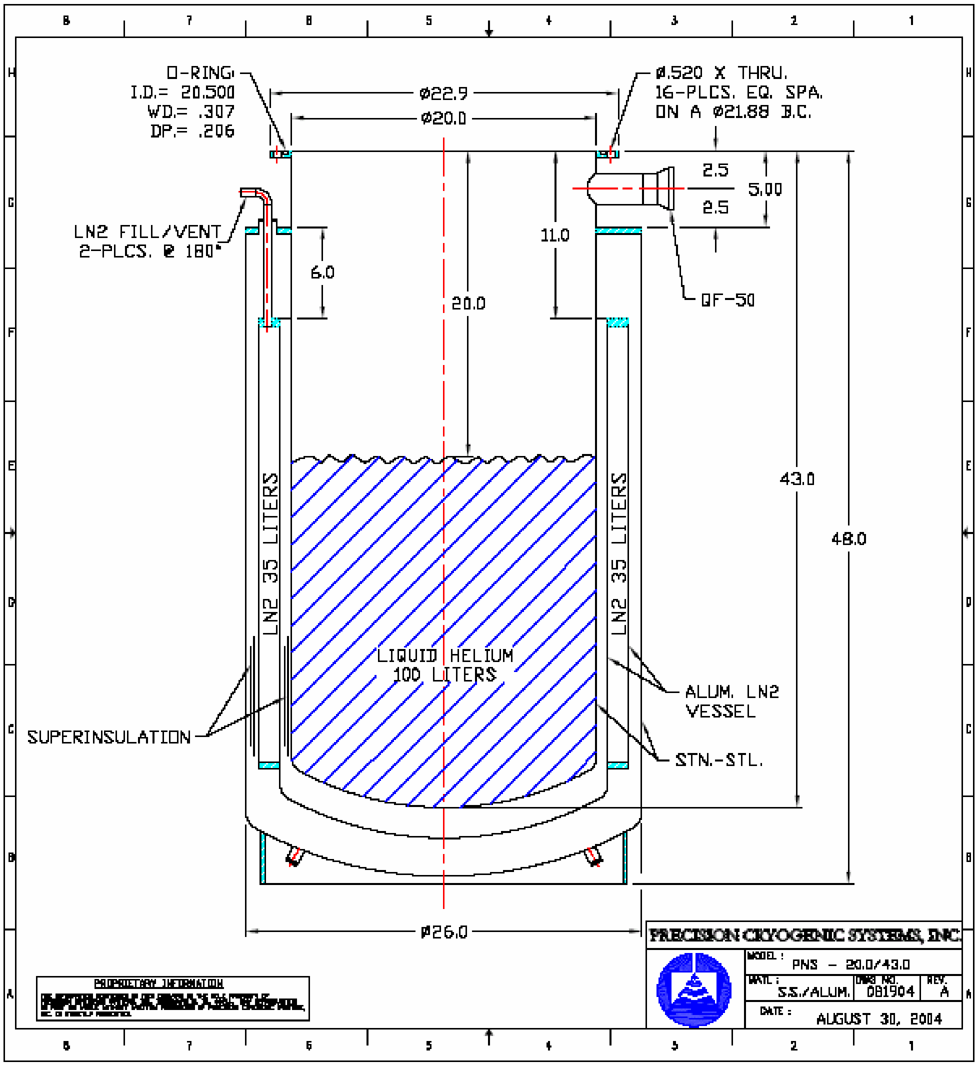


Figure 18: Drawing of RRR measurement cryostat.

4) Appendix

4.1) Lab-Layout & Floorplan

4.2) Chemistry Hood Memo

4.3) ODH Analysis

4.4) Electrical Plan

4.5) Duct Dimensioning

4.6) Chemical Safety Analysis

4.7) Chemical Hygiene Plan

4.8) Pump Memo

4.9) Pump Calculation

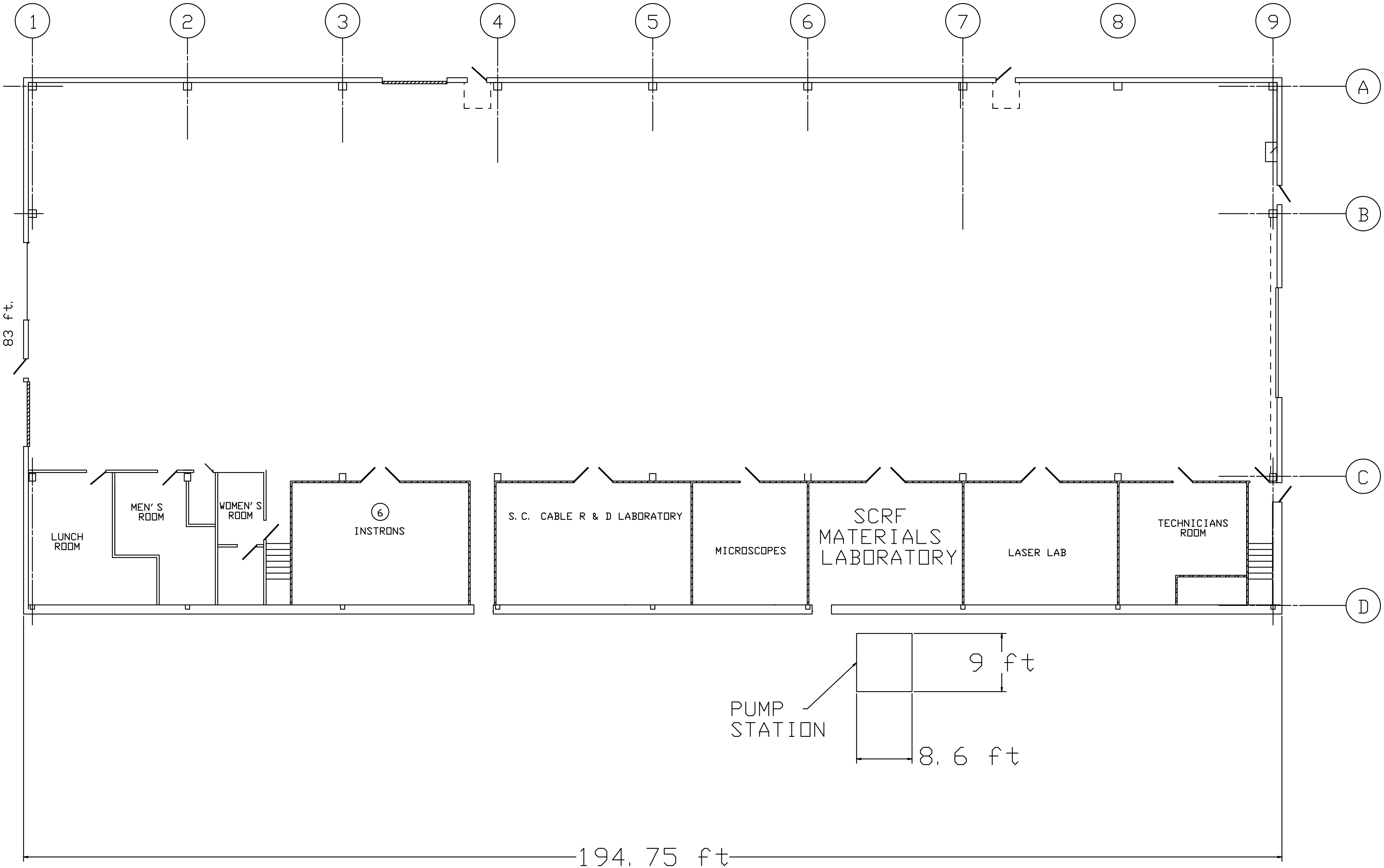
4.10) Heat Exchanger Calculation

4.11) Pressure Vessel Safety Note

5) Glossary

BCP	Buffered chemical polishing
EP	Electropolishing
LN	Liquid nitrogen
MDL	Material Development Laboratory
RRR	Residual resistivity ratio
SRF	Superconducting radio frequency

APPENDIX 1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4
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6
7
8
9
A
B
C
D
83 ft.
LUNCH ROOM
MEN'S ROOM
WOMEN'S ROOM
6
INSTRONS
S. C. CABLE R & D LABORATORY
MICROSCOPES
SCRF MATERIALS LABORATORY
LASER LAB
TECHNICIANS ROOM
9 ft
PUMP STATION
8.6 ft
194.75 ft

APPENDIX 2

P. Bauer / D. Hicks / Y. Terechkine – Fermilab/TD

09/13/2004

Memo regarding chemistry hood for IB3-SRF Materials Lab

In the frame of recent efforts at Fermilab to develop expertise, infrastructure and a technology base for SRF technology, a new SRF materials lab was recently created. One of the systems that need to be installed in this new lab space is a chemistry bench for the preparation of small material samples.

It is common practice to install such chemistry benches in material test labs because it allows for fast turn-around in sample preparation. It is also being used for chemical surface preparations of samples in cases in which the delay between surface treatment and testing needs to be minimal. Finally such a system is also convenient for the simple cleaning of samples.

We understand that it is Fermilab policy to pool as much as possible chemistry facilities to reduce the risk of accidents involving acid spills, ...etc. We also need to point out that there currently is a chemistry facility in the Material Development Lab (MDL) in the village where sample preparation tasks are performed. Finally, an additional factor that needs to be factored into the equation is, that in ~2.5 years the MDL facilities will be moved to the new IB3 extension and therefore a close-by chemistry facility will exist after that.

Installing a chemistry facility in the SRF materials lab in IB3 is therefore a contentious issue, which needs to be resolved before we can move on. We would like to install such a system in order for the SRF materials lab to operate efficiently now. This installation includes a blower and hood, which collect and direct chemistry fumes out from the lab through a chimney that reaches ~10′ above the roof of the IB3 building. The chemistry hood would also include an acid storage cabinet with small quantities of acids, as needed for the etching and polishing of small samples of SRF materials. These processes typically involve nitric, sulphuric, phosphoric and hydrofluoric acids.

With the start of operation of the IB3 extension and the ensuing close proximity of the new MDL the chemistry hood in the IB3 SRF materials lab would become obsolete. For this case the hood facility would be dismantled. The possibility of a transfer of the equipment to MDL or another facility (e.g. clean-room facility in IB4) should be envisaged.

Summarizing the above said, this memo serves two purposes: -1- to indicate our interest to install a small chemistry facility to be used for a chemistry on small samples in the SRF materials lab in IB3 and –2- our promise to remove this facility whenever a close-by MDL becomes operational.

APPENDIX 3

Tom Peterson, February 2005

Helium and Nitrogen ODH Analysis for IB3 SRF Materials Laboratory

<u>References:</u>

Fermilab ES&H Manual, Chapter 5064, "Oxygen Deficiency Hazards".
"Cryogen Usage in the Engineering Laboratory", memo from W. Boroski to Tom Peterson, April 2, 1991.
Sketch of SRF Materials Lab floor plan, by G. Lorenz, 2004.
"Safety in Handling Helium and Nitrogen", by G. Schmauch, et. al., of Air Products and Chemicals, Inc., 1991, presented at the IISSC-3 conference in Atlanta.

<u>Introduction</u>

The SRF Materials Lab in the Industrial Building 3 uses small volumes of liquid helium and liquid nitrogen for testing of SRF materials at low temperature. The liquid helium is provided via transfers from 500 liter commercial helium storage Dewars, and the liquid nitrogen from 150 liter commercial Dewars. This analysis examines the oxygen deficiency hazard due to a release of cryogens in the SRF Materials Lab.

This type of handling and transfer of cryogens in a room is similar to that done for MRI systems and analyzed in the paper by Air Products and Chemicals, Inc., referenced above. Although my analysis is done entirely in accordance with the requirements of Fermilab's ES&H manual, some numbers in the Air Products paper are referenced for comparison. In every case Fermilab's standard is the more conservative.

<u>Room Data</u>

Engineering Lab floor area (= ceiling area) = 19x24 sq.ft. = 456 sq.ft.
Ceiling height = 9 ft.
Room volume = 4104 cubic feet.

<u>Helium ODH Considerations</u>

The maximum liquid helium container volume is 600 liters. If released and warmed to room temperature this would fill 15960 cubic feet, approximately three times the room volume. If mixed with the remaining air in the room, the concentration of oxygen would be 5%.

The release of the entire contents of a 500 liter helium Dewar results in a fatality. (It is always full and all released.) The release of the content of a Dewar can be caused by the rupture of the Dewar insulating vacuum or that of any vessel or line attached to the Dewar. It can also be caused by a "large event" during a U-tube (transfer line) change.

From Table II in the ES&H manual chapter 5064TA we have the following relevant probabilities:

Dewar leak or rupture	1×10^{-6}/hr
Cryogenic fluid line leak or rupture	3×10^{-6}/hr
U-tube (transfer line) change release, large event	1×10^{-3}/demand

The lab could have as many as one 500 l helium Dewar and a 100 l cryostat (helium reservoir) in it at once, possibly connected to each other. The cryostat is not yet designed. Possibly, however, it can be designed such that its safety valve connects to an external vent-line. In this case the cryostat in the stand-alone mode would not be of concern here. In the worst case the cryostat is connected to the 500 l Dewar. To represent this worst case scenario I will count them together as one 600 l system. Counting them as two reservoirs the total probability of a Dewar leak or rupture is 2×10^{-6}/hr. The system includes a transfer line to the cryostat (reservoir) and a vent line from the reservoir, for a total of 2 lines connected to a total of 600 liters, so the total probability of a cryogenic fluid line leak or rupture is 6×10^{-6}/hr. The 41 LHe transfers per year entail 82 U-tube operations per year (counting stinging and removing as separate). So the total probability of a release (large event) during a U-tube change is 84×10^{-3}/year = 9.6×10^{-6}/hr.

The total probability of the release of the entire contents of a 500 liter helium Dewar and a 100 l cryostat is $(2+6+9.6)\times10^{-6}$/hr = 1.76×10^{-5}/hr. Since a fatality factor of 1 has been assumed for these events, the ODH hazard class would be 2 (although close to the lower bound). But now consider ventilation.

Suppose a vent fan is connected to an ODH sensor, which is fail-safe, but the vent fan is like a pump in that it fails to start 1×10^{-3}/demand. (The safety manual has no statistics explicitly for fans, but I would expect it to be electrically like a pump and mechanically more reliable than a pump, so this is conservative.) If this vent fan is sized to prevent the helium from accumulating in the engineering lab in the event of a 600 liter release, then only when there is a release AND the fan fails to run is there a fatality. The probability of this occurring is $(1\times10^{-3})\times(1.76\times10^{-5}$/hr) = 1.76×10^{-8}/hr. This situation is now ODH 0.

<u>Helium Release Rate and Required Fan Size:</u>

The maximum flow rate from a 500 liter helium storage Dewar would result from a loss of insulating vacuum to air and the resultant air condensation on the inner vessel. The venting could occur from a relief valve or another vent valve on the vessel if it happened to be open. I will assume that the reliefs are set and sized to prevent the vessel from exceeding 15 psig in the worst case, so that the vessel is not considered to fall under the scope of the ASME pressure vessel code (industry interprets the Code's "15 psi" as 15 psig, rather than 15 psid as does Fermilab). I have tried to obtain information from AirCo regarding the sizing of their relief valves but have not succeeded, so I will use this assumption and back-calculate a flow rate.

The AirCo 500 liter helium Dewars delivered to the SRF Materials Lab have two Circle Seal Relief valves, each #559B-6M, one set at 8 psig and one at 10 psig. From the Circle Seal literature, the 6M valve cracking at 8 psig will relieve 60 SCFM air, or 171 SCFM helium, at 14 psig. The 6M valve cracking at 10 psig will relieve 40 SCFM air, or 114 SCFM helium. With a loss of vacuum the large flow rate of helium will result in temperatures much lower than room temperature, close to 5 Kelvin. A helium temperature of 20 K is conservatively warm and also enough in the ideal gas range for helium that the flow calculation for gas is still accurate. Scaling the flow with the square root of density from 273 K to 20 K gives flow rates of 633 SCFM for the 8 psig relief and 421 SCFM for the 10 psig relief. The total flow rate is 1054 SCFM out of the reliefs with 14 psig dewar pressure.

The 100 l liquid helium cryostat is not yet designed. We will therefore seek to use safety valves of comparable design as above and use a direct vent-line to the outside to prevent the release of helium from the cryostat into the laboratory. There is still, however, the possible transfer of gas from the 100 l cryostat to the 500 l Dewar through the transfer line. In the case of a large event in the cryostat a similar event in the Dewar would therefore ensue and the above estimation applies in this case.

To check the maximum flow that could be pushed out of a transfer line, use the 0.21 inch measured stinger inner diameter, and suppose liquid is driven out with a 1 atmosphere differential pressure. The result using the Fike rupture disk liquid formula for a 0.21 inch diameter hole (neglecting viscous pressure drop in the line), 14 psig pressure driving the liquid, and 0.125 g/cc liquid density, is 801 SCFM of helium.

So the case of gas venting from the reliefs, 1054 SCFM helium, is the greatest flow. 1054 SCFM of helium is 88 grams/sec or 42.3 liters/min, so a full Dewar empties in about 12 minutes.

One air change per hour would be 4560 cubic feet per hour or 76 CFM. 1054 CFM would be 14 air changes per hour. My experience tells me that the helium will not mix with the air but will rise to the top of the room. So either a minimum of 1054 CFM (14 air changes per hour) venting from the top of the room should be continuously provided or this amount of venting should be triggered by the ODH sensor.

Comparison to Air Products MRI Analysis:

The paper "Safety in Handling Helium and Nitrogen", referenced above includes an analysis of a situation similar to that of our SRF Materials Lab. In particular, a few numbers may be compared to those in this analysis.

The vent rate for "Dewar vacuum loss" of a 500 liter helium storage Dewar is 1036 SCFM and is the largest vent rate. (This is to be compared to the 1054 SCFM that I calculated.) The other large vent rates from the helium Dewar result from "transfill hose failure" (332.8 SCFM) and "V-2 venting" (422.67 SCFM). The total of the probabilities given for these three events is 4.15×10^{-5}/year, almost completely dominated by the Dewar vacuum loss probability. This is 4.74×10^{-9}/hr, about a factor of 1000 less than what our ES&H manual provides. I believe this indicates that our initial estimates of failure rates, still in our ES&H manual, are much more conservative than what industrial experience indicates.

Nitrogen ODH Considerations:

By far the largest single nitrogen volume (see Bill Boroski's "Cryogen Usage . . . " memo, April 2, 1991) is that of the 150 liter storage Dewar, so consider a release of the entire contents of a 150 liter LN2 Dewar. 150 liters warmed to room temperature at one atmosphere would result in 3690 cubic feet of nitrogen. If this were slightly cool and covered the 456 square foot floor, it would fill the entire lab. This has a fatality factor of 1 according to Figure 3 of 5064TA in our ES&H manual. I will use this as the fatality factor for a release of LN2.

Our ES&H manual does not distinguish between helium and nitrogen Dewars or lines in the table of probabilities, so we assume the same probabilities as above. There are no U-tube changes for LN2, but transfers are made through foam-insulated tubing. I will treat those transfers as if they were being done through a U-tube, although the operation is safer than that, in order to get an estimate for the probability of a major release. So suppose the probability of a major release is 1×10^{-3}/demand. Bill Boroski reports 81 transfers during 1990, which, taking this to be a typical year, and saying a failure can occur during connect or disconnect, as for helium, gives $2\times81\times10^{-3}$/yr = 1.8×10^{-5}/hr.

There is at most one Dewar at a time in the SRF Materials Lab. The total probability of a Dewar leak or rupture is therefore 1×10^{-6}/hr. A line from the Dewar to an experimental apparatus would result a leak or rupture probability of 3×10^{-6}/hr. The total probability of a major release of N_2 is then $(18+1+3)\times10^{-6}$/hr = 2.2×10^{-5}/hr. The probability of a fatality due to a release of LN2 is 2.2×10^{-5}/hr $\times$ 1×10^{-3}/event = 2.2×10^{-8}/hr. This is much less than 10^{-7}, resulting in ODH 0 for nitrogen.

Conclusions

Either a minimum of 1054 CFM (14 air changes per hour) venting from the top of the room to prevent helium accumulation should be continuously provided or this amount of venting should be triggered by an ODH sensor in order to have an ODH 0 rating in the SRF Materials Lab. The small amounts of LN2 present do not present a significant hazard.

SUBJECT

IB3 DP18 –Electrical/ODH

NAME
Jim Garvey

DATE
02/02/2005

REVISION DATE

The new material development lab (SCRF) in IB3 will need to address several electrical issues and one ODH hazard abatement. Present plans call for the installation of a vacuum pumping station with a 10 HP Roots Blower motor, a 25 HP Second Stage motor, and a 2.0 kVA control transformer for the motor starters. Due to noise, vibration, and heating, the vacuum motors will be placed in a sound shielding enclosure outside the lab adjacent to the building.

Additionally there will be a chemistry hood installed in the same room, which must have a dedicated "GFCI" circuit for the eye wash station, and separate 110v circuits for the sump pump and lighting.

At the far northeast corner of the lab, work will be done with H_e Dewar requiring a dedicated circuit for ODH monitoring, signaling, and damper operation.

Electrical:

. Vacuum Pumping Station (Fed from *DHP-IB-VII-2 Circuit # 6*)

- 10 Hp Roots Blower @ 480 volts — 14.67 Amperes
- 25 Hp Second Stage @ 480 volts — 35.48 Amperes
- 2.0 kVA Control Transformer @ 480 volts — 4.17 Amperes

<54.26 Amperes> FLC

Allowing 125% for CB Use **HFB 100 Amperes**

.Single drop fusing

- F1 (10Hp) 14.67 * 1.73 = 25.38 — DEF 25 Ampere Fuse
- F2 (25Hp) 35.48 * 1.73 = 61.38 — DEF 70 Ampere Fuse
- F3 (2.0Kva) 4.17 * 1.8 = 7.5 — DEF 10 Ampere Fuse

10Hp & 25Hp motors shall use Square D Nema2 enclosures for Fused Safety Disconnects.

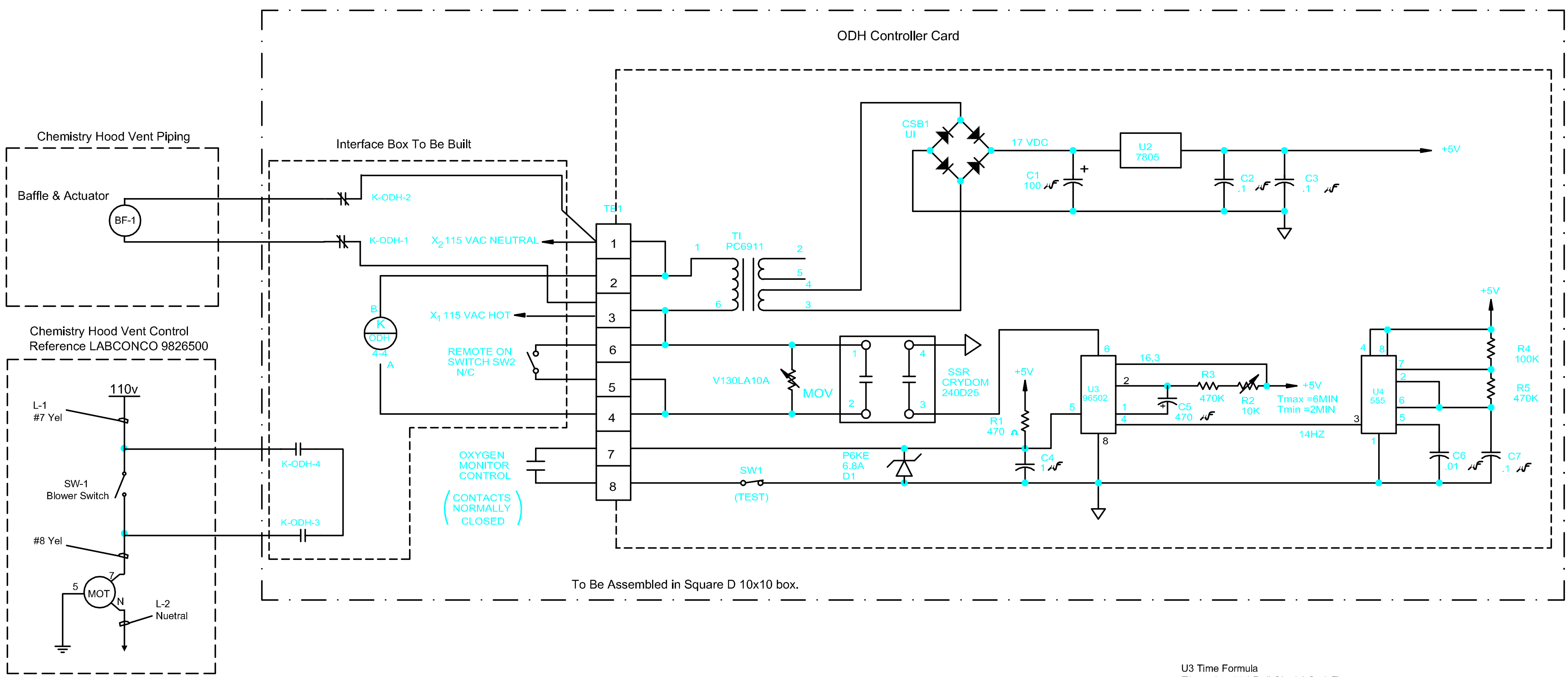
. Chemistry Hood

- 120v/ 20 Amp circuit for blower motor.
- 120v/ 20 Amp circuit for service lighting.
- 120v/ 20 Amp circuit for optional outlet receptacles.
  The above will be provided by one 120v, 3phase w/ neutral & ground fed from PP-IB-VII-3 ckt 38,40,42
- 120v/ 20A GFCI protected receptacle located at eye wash station, PP-IB-VII-3 ckt 21
- 120v/ 20A receptacle for sump pump at bottom of hood, PP-IB-VII-3 ckt 23

ODH Abatement:

In concert with Engineering Note issued by Tom Peterson dated 02-05, there will be a sensor mounted high in the room for Helium leaks and another mounted low for Nitrogen leaks. Both sensors are connected to an ODH Control Chassis with horn annunciation and strobe lights. Venting of the area will be use of a damper control mounted in series with the chemistry hood vent piping. Operation of the damper will be controlled by the "motor control card" of the ODH unit (see sketch JDG00020305) and a Honeywell model ML4125 actuator, with positive spring control to "open" position in the event of loss of power. Calculations required a vent rate of 1054 CFM minimum; therefore the original motor of the chemistry hood vent was replaced with a .75 Hp unit rated at 1400 CFM (model 7181400). This reduced the need to puncture another hole through the sidewall and has the added advantage of disbursing Helium high above the normal walk area of personnel.

To maintain "fail safe" mode of operation, the ODH control logic will be reversed at the rear connector (J3) of the control chassis. This system shall be fully tested prior to any work commencing in the area. In addition, CFM rates of the venting system will be verified and documented as well. Dedicated circuit for this unit will be 120v/20A PP-IB-VII-3 circuit # 12.



U3 Time Formula
T(msec) = .55 * Rx(kOhm) * Cx (uF)
Rx = R2 + R3 (2Mohm max.)
Cx = C5

IB3 SCRF Lab ODH Control	Drawn By: Jim Garvey Checked: Approved:	Date: 2/10/05 Rev.: A	Fermi National Accelerator Laboratory United States Department of Energy Technical Division JDG00021005

APPENDIX 5

SRF Materials Lab IB3
VENTILATION SYSTEM
C. Boffo Sept. 2005

<u>*System description:*</u>

The ventilation system of the SRF Materials lab is designed to:

- Extract the fumes from the chemical hood
- Be part of the ODH system

The design flow rate of 1054 SCFM is defined by the ODH requirements [Appendix 3 and 4], which are more demanding with respect to the hood fume extraction. The air speed in the duct should be kept in the 2000 fpm range to avoid condensation especially in the horizontal legs of the system. The duct must be made in PVC to avoid corrosion due to the chemical species extracted (HNO_3, HF, H_2SO_4, H_3PO_4) and is routed to the IB3 roof inside the building for aesthetics. The PVC capability to withstand contact with concentrated quantities of the anticipated mixes of acids to be used in the hood has been tested at Fermilab, moreover the diluted fumes are less aggressive compared to the concentrated acids. The fan (fiber glass) is placed on the roof so that the duct inside the building is kept below atmospheric pressure. This is the safest solution for personnel since in case of small leak the fumes do not leave the duct. The rectangular duct must be fabricated with double welded parts. All connections between round piping and rectangular duct must be sealed. The fan and chimney are placed at the maximum distance possible from any existing air intake of the building and moreover the exhaust stack height allows minimizing the possibility of the fumes to circulate back into the building.
In accordance with NEC 250.190, the zero pressure weather extraction stack is connected to the roof girders in four points which also serve as lightning grounding. The changes in directions are obtained using sweeping elbows or using custom boxes to reduce losses where possible.

The system can be divided in three parts:
1. 12″ duct inside the Lab
2. 5″x20″ rectangular duct inside IB3
3. 10″ duct on the roof

1. The part in the lab is 12″ in diameter to reduce the friction losses. In this area there are 3 elbows and one tee that are the most significant sources of losses in the ducts. The duct is above the false ceiling for aesthetics and two drop points are visible one for connection to the hood and the other for the ODH system on top of the cryostat. The larger diameter reduces the air velocity but being the room heated in winter and being a rather short circuit the condensation of chemical should be very limited. The horizontal part is tilted up 1 degree toward the connection with the rectangular duct where a capture area with an opening is setup to collect condensation fluids. The pipe for the ODH system is tilted as well to avoid condensed fluids from dripping on top of the cryostat. One actuated damper is used to switch between the hood flow and the ODH system flow.

2. The part in the IB3 hall is rectangular to fit between the mezzanine wall and the crane columns. The office walls serve as a perfect structural support for the duct that can shoot straight to the roof where the penetration to the outside is anticipated.

3. The part on the roof is 10" in diameter to maintain high air speed and avoid condensation even in cold environment. The duct connects to the fan which also serves as a base for the extraction stack for a total height of ~7'. The chimney is placed ~36' away from the other air intakes of the building HVAC system and is supported in four points which also serve as electrical grounding.

The make up air will be provided through an opening in the lower area of the lab's doors.

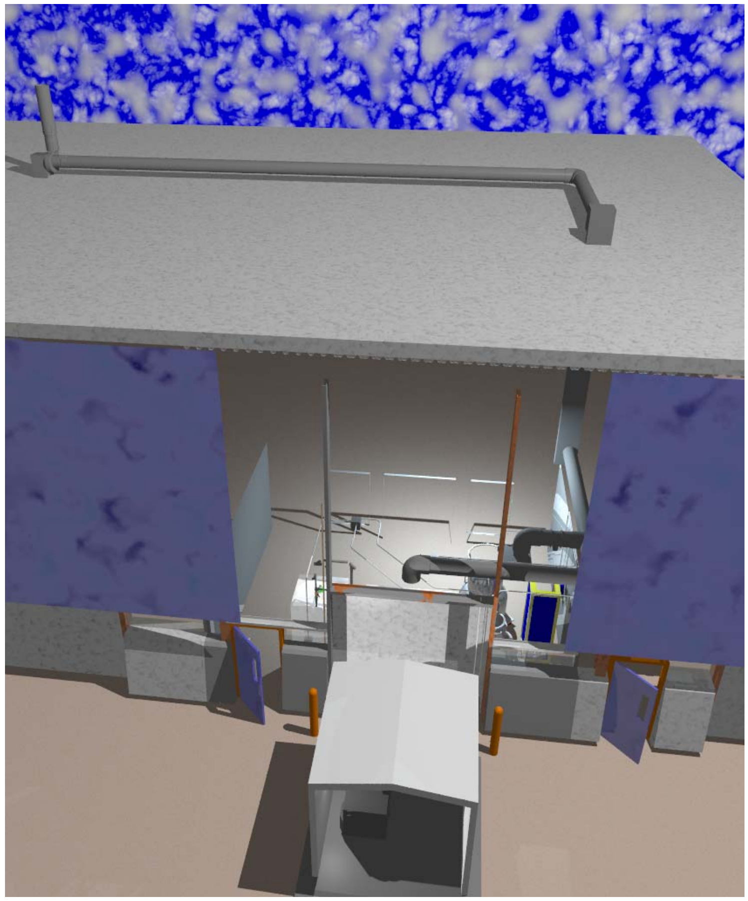
Calculations:

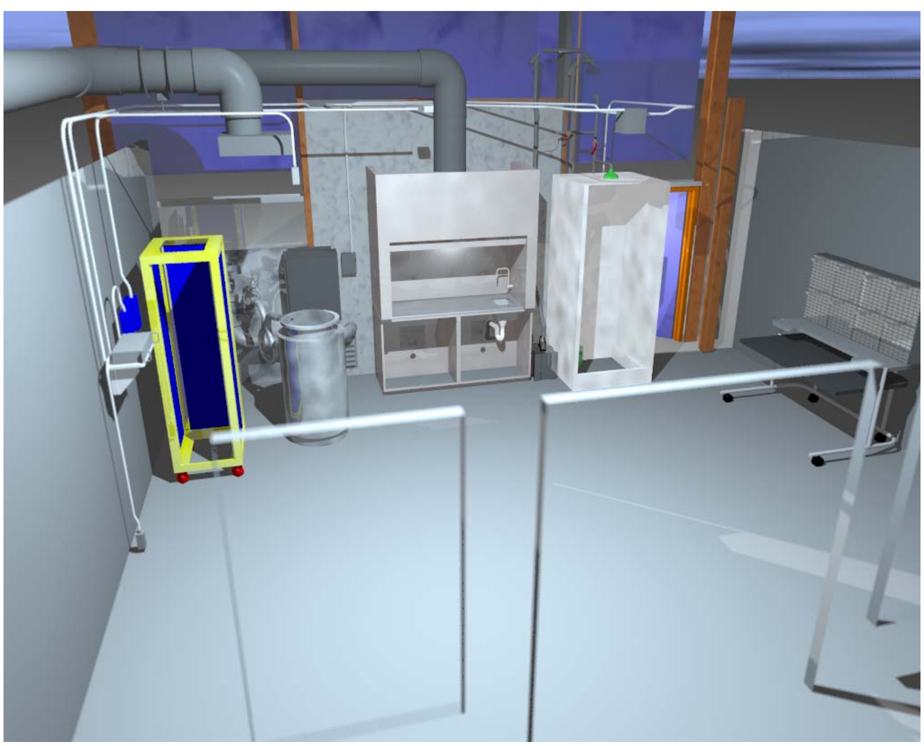
According to this table below the loss related to the straight piping is 0.556 inches of water. To this number one must add the pressure drop related to elbows and chimney. These concentrated losses are calculated as additional straight pipe in the system with coefficients provided by the duct vendors. Adding these two lengths the total pressure drop in the system is 1.200 inches of water (311 Pa). At the maximum speed of 1300 rpm the fan can provide up to 1.200 inches of water at 1100 CFM meeting the system requirements.

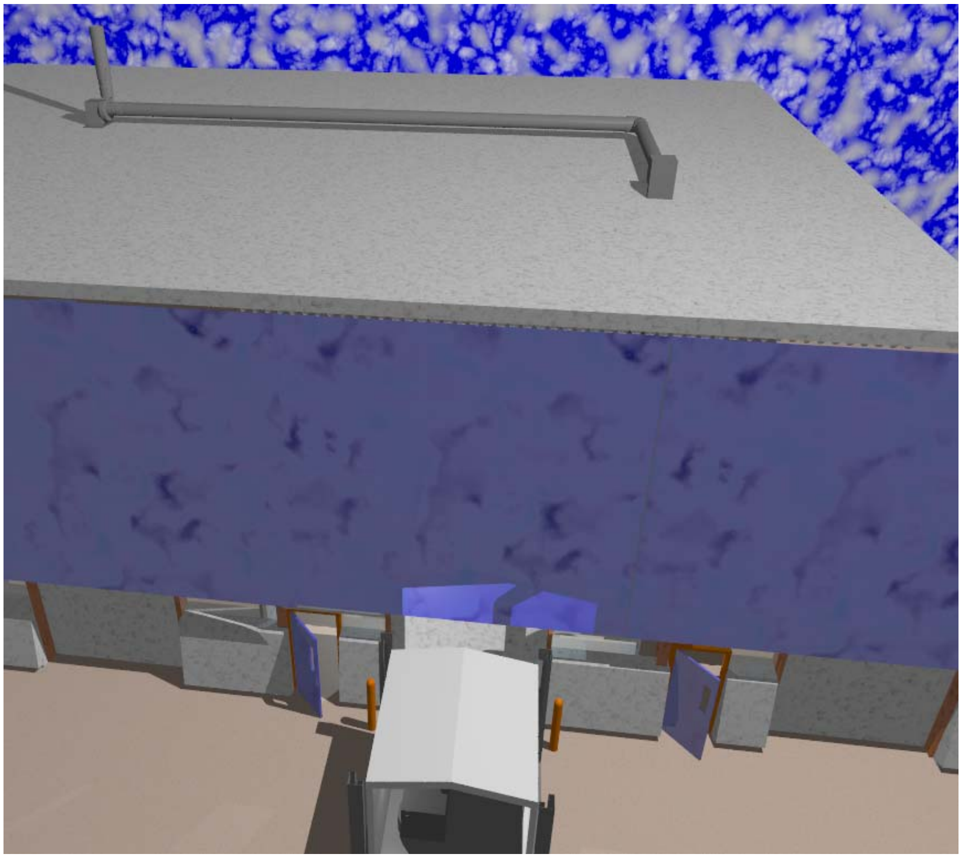
Duct pressure drop and fan dimensioning:

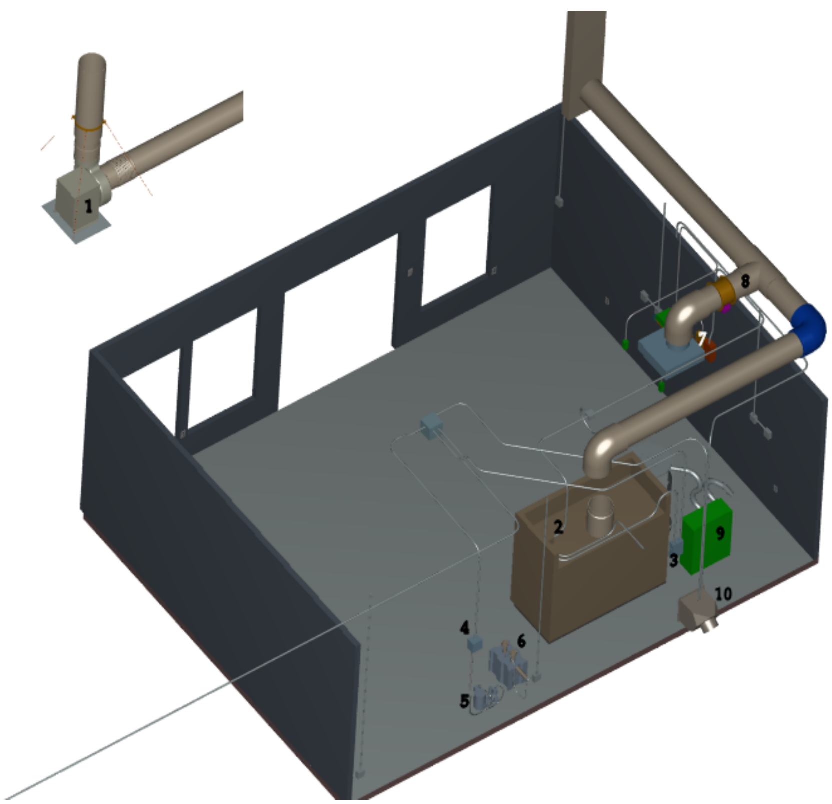
Flow rate	Q = 1100 cfm
Average speed	V = 2000 fpm
Diameter of circular pipe	d
Rectangular duct dimensions	a and b
Equivalent diameter	deq = 1.3* (a*b)^0.625/(a+b)^0.25
Duct cross section	A
Specific friction loss [2]	dPspec
Correction of friction between galvanized and PVC ducts k	
Pressure drop	DP

Zone	d	a	b	deq	A	V	dPspec	k	Length (*including elbows & tees)	DP
	in	in	in	in	ft2	fpm	inH2O/ 100ft		ft	inH2O
1	12				0.785	1401	0.3	0.94	16.45 / 75.7*	0.046 / 0.21*
2		20	5	10.34	0.583	1887	0.6	0.94	19.74	0.111
3	10				0.545	2017	0.6	0.94	59.21 / 141.5*	0.334 / 0.798*









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APPENDIX 6

Chemical Safety Analysis

P. Bauer, C. Boffo, D. Hicks

February 2005

<u>Chemicals</u>

Hydro-fluoric acid, HF is a colorless, fuming liquid or gas (depending on the temperature) with a strong, irritating odor. It is solvable in water and boils at 20°C/1atm. It is non-flammable but can produce explosive hydrogen gas in conjunction with moisture or steam. The air odor threshold for hydrogen fluoride is 0.042 part per million (ppm) parts of air. The current Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for hydrogen fluoride is 3 ppm as an 8-hour time-weighted average (TWA) concentration [29 CFR 1910.1000, Table Z-2]. Inhalation of hydrogen fluoride gas may cause immediate or delayed onset pulmonary edema after a 1-hour exposure. The lowest lethal concentration for a 5-minute human exposure to hydrogen fluoride is in the range of 50 to 250 ppm. Skin contact with hydrogen fluoride or solutions containing more than 30 percent hydrogen fluoride produces immediate pain; reactions to more dilute solutions may be delayed for many hours. The accompanying pain is excruciating and persistent, and healing is delayed. Severe eye injuries may occur from splashes. Liquefied hydrogen fluoride gas has been known to destroy the eye and to require enucleation; the severity of burns from the aqueous solution depends on the concentration. Ingestion of an estimated 1.5 grams of hydrofluoric acid produces sudden death; repeated ingestion of small amounts of hydrogen fluoride may cause fluoride osteosclerosis. Hydrogen fluoride should be stored in a cool, dry, well-ventilated area in tightly sealed containers that are labeled in accordance with OSHA's Hazard Communication Standard [29 CFR 1910.1200]. Containers of hydrogen fluoride should be protected from physical damage and should be stored separately from metals, concrete, glass, strong bases, sodium hydroxide, potassium hydroxide, and ceramics.

Nitric Acid, HNO_3, is a colorless liquid with a sharp pungent odor. It should not be in contact with strong bases, metallic powders, carbides, H_2S, and combustible organics. Conditions to avoid are high heat, open flames and other sources of ignition. When heated to decompositon it emits toxic oxides of nitrogen fumes (NO_x). The current Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for Nitric acid is 2 ppm as an 8-hour time-weighted average (TWA) concentration [29 CFR 1910.1000, Table Z-2] or $5mg/m^3$. Inhalation can cause coughing, choking, and inflammation of the respiratory tract. Swallowing can cause burns to mouth and g.i. tract.

Eye contact can cause severe burns and damage. Skin contact can cause severe burns. The chronic effects of concentrated vapors can be bronchitis and erosion of teeth. The acid should be stored in a cool, dry, well ventilated area. The containers should be tightly closed when not in use. The containers should be protected from physical damage and direct sunlight. A small spill can be flushed with water and neutralized with alkaline material (sewer neutralized material with excess water). Following a large spill, the area needs to be evacuated and ventilated. If possible, the leak should be stopped and dikes used to retain run off. Large spills can be neutralized with soda ash, lime or other suitable alkaline material.

Nitrogen Dioxide, NO_2, is a reddish gas with a pungent, irritating or suffocating odor. It can be severely irritating in the presence of moisture. Over-exposures to this gas mixture may result in severe irritation of eyes, skin, mucous membranes, and any other exposed tissue. If exposure is prolonged, delayed pulmonary damage and breathing difficulty may occur. Severe over-exposures can be fatal. This gas mixture is not flammable or reactive. It is stable at standard temperatures and pressures. The current Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for Nitrogen Dioxide is 5 ppm as an 8-hour time-weighted average (TWA) concentration [29 CFR 1910.1000, Table Z-2] or 9 mg/m_3. Over-exposure to this gas mixture may result in severe irritation of eyes, skin, mucous membranes, and any other exposed tissue. Prolonged exposure to this gas mixture may cause more noticeable effects in persons with pre-existing lung disorders such as asthma. Prolonged or repeated over-exposures to this gas mixture may cause impaired lung function and other adverse respiratory tract symptoms. Prolonged skin exposure may cause dermatitis. The respiratory system, skin, eyes are the target organs of acute exposure. Skin, the respiratory system and immune system are the target areas of chronic exposure.

The BCP acids are delivered by a vendor and stored in 2.5 or 4 lit bottles made or lined with a Polytetrafluoroethylene (PTFE, e.g. TEFLON) material. The bottles must be periodically replaced. All containers used to contain the BCP acid mix must be made of Polyvinylidene Fluoride (e.g. PVDF) that is rated for use with the three acids. During loading and unloading, personal protective clothes must be worn. Acid must be stored in a cool and well-ventilated area and bottles must be tightly closed. While opening barrels for use, personnel must wear protective clothes and use local ventilation to prevent exposure to acid fumes.

Process Parameters

During the chemical polishing process five moles of NO_2 evolve for each mole of Nb. The surface of Nb exposed to acid in the SRF Materials Lab is always going to be $<1ft^2$. The maximum etching depth for one etching cycle is about 0.004 inches. This results in a total volume of removed Nb of $0.5\ in^3$ or 0.15 lbs that corresponds to <1 mole. Therefore
A maximum of five moles of NO_2 will evolve during an etching session (0.4 lbs). Taking into account an etching rate of about 0.00004 in/min at 59 F, the total etching cycle will take 100 minutes, and gas release rate will be about 0.3 lbs/h. This is 350 times the TWA concentration of 9 mg/m^3.

Mists of hydrofluoric and nitric acids evolve during the process. The evaporation rates can be calculated using of the Evaporation Calculator available from a well-known web site[1]. The amount of mists depends on the stage of the process, its temperature and ventilation conditions. The evaporation rate for HF is 0.00125 lbs/h per 1 sqft of open acid surface. Assuming a maximum open surface of 1sqft, so RHF = 0.00125 lbs/hour, ~2 times the TWA limit after one hour. The evaporation rate for HNO_3 is 0.002 lbs/h per 1 sqft of open acid surface. Assuming a maximum open surface of 1sqft, so HNO_3 = 0.002 lbs/hour, ~3 times the TWA limit after one hour.

[1] http://response.restoration.noaa.gov/

Neutralization

A neutralization process is used to bring the pH of used water to the allowable limit of about 5.5 –9, before discharge through the public sewer. This procedure only applies to rinsing water and small spills (see “chemical hygiene” document for further details).

CHEMICAL HYGIENE PLAN

MATERIALS DEVELOPMENT and TESTING LABORATORY (MDTL)

SUPERCONDUCTING RF (SRF) MATERIALS LABORATORY

TECHNICAL DIVISION

Revision 4.0
April 11, 2005

DIVISION APPROVALS

Technical Division ES&H Group Richard Ruthe 4/12/05
Richard Ruthe, CIH, SSO Date

MDTL Chemical Hygiene Officer Charlie Cooper 4/12/05
Charlie Cooper Date

SRF Chemical Hygiene Officer Cristian Boffo 4/12/05
Cristian Boffo Date

Engineering & Fabrication Apollinari 4/27/05
Giorgio Apollinari, Department Head Date

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D. STANDARD OPERATING PROCEDURES

Appendix A

A. GENERAL PRINCIPLES

1) Introduction

Very few laboratory chemicals are without hazards. It is prudent to minimize employee exposure by taking precautions for handling these chemicals. The purpose of this Chemical Hygiene Plan is to ensure that workers in the Materials Development and Testing Laboratory (MDTL) and SRF Materials Laboratory are protected from health hazards associated with the various chemicals with which they work. The Chemical Hygiene Plan is part of compliance with the regulations promulgated on January 31, 1990 by the United States Department of Labor Occupational Safety and Health Administration (OSHA) entitled "Occupational Exposures to Hazardous Chemicals in Laboratories" (29 CFR 1910.1450). The Chemical Hygiene Officer and the Technical Division ES&H Group will review this Chemical Hygiene Plan annually.

The OSHA Laboratory Standard only applies to laboratory workplaces where chemicals are used in a nonroutine, nonproduction manner by workers with at least some education and training in science. Laboratory use of hazardous chemicals is defined as the handling or use of hazardous chemicals in which all of the following criteria are met:

- Procedures using chemicals are carried out on a laboratory scale (e.g., using containers for reactions, transfers, and other handling of chemicals that are easily manipulated by one person).

- Multiple chemical procedures or chemicals are used.

- The operations involved are neither part of a production process nor simulate one.

- Protective laboratory practices and equipment are available and are commonly used to minimize the potential for employee exposure to hazardous chemicals.

When the operations in a particular facility meet all of the above criteria, that facility must comply with the requirements of this Chemical Hygiene Plan. Operations in facilities involved in the use of hazardous chemicals but do not meet the criteria previously outlined shall comply with the health hazard communication program including all other applicable OSHA regulations.

2) Applicable Standards

Title 29 Code of Federal Regulations 1910.1450.

3) Minimizing Chemical Exposures

Exposure to chemicals will be minimized even for substances with no known significant hazard. For those substances that present special hazards, special precautions will be taken.

4) Permissible Exposure Limits and Threshold Limit Values

The MDTL and SRF Materials Laboratory recognize and will not exceed the Permissible Exposure Limits of OSHA or the Threshold Limit Values of the American Conference of Governmental Industrial Hygienists. This will be accomplished by performing their hazardous chemical operations within the chemical fume hoods.

B. CHEMICAL HYGIENE RESPONSIBILITIES

1) Technical Division Head

The Technical Division (TD) Head has the ultimate responsibility for chemical hygiene within the MDTL.

2) TD Engineering & Fabrication Department Head

The TD Engineering & Fabrication Department Head has administrative responsibility for chemical hygiene within the MDTL.

3) Chemical Hygiene Officer

The Chemical Hygiene Officer is responsible for assessing the hazards associated with all work to be performed in their designated laboratory and providing the adequate safeguards. All experimental procedures must be reviewed by the Chemical Hygiene Officer before they are implemented to assure work is conducted and is appropriate to the physical facilities.

The Chemical Hygiene Officers must assure that all employees have been adequately trained and are following safe work practices.

Charlie Cooper is the Chemical Hygiene Officer for the MDTL.

Cristian Boffo is the Chemical Hygiene Officer for the SRF Materials Laboratory.

4) Environment Safety and Health (ES&H) Group

The TD Environment Safety and Health Group will assist in detecting hazardous operations and conditions, establishing safe work practices, and selecting special protective equipment.

5) MDTL Employees and Other Users

MDTL employees and those outside TD using MDTL must conduct each operation in accordance with the Chemical Hygiene Plan.

6) SRF Materials Laboratory Employees and Visitors

Authorized SRF employees and authorized visitors must conduct their activities in accordance with the Chemical Hygiene Plan.

C. GENERAL PRINCIPLES

1) Policy

It is the policy of the Technical Division to provide employees working in the MDTL and SRF Materials Laboratory with a safe and healthful working environment. This Chemical Hygiene Plan (CHP) outlines the manner in which this is accomplished. It is written in accordance with the OSHA standard 29 CFR 1910.1450.

2) Chemical Procurement

The ES&H Group performs ES&H and NEPA reviews on all requisitions. This will serve as a notice that a new chemical is being added to the inventory. If a particularly hazardous chemical is being considered, the ES&H Group will be notified before the requisition is submitted and supplied with a current MSDS for the chemical. The ES&H Group will review the proposed usage and safeguards before the chemical can be ordered.

No chemical shall be accepted unless the container is adequately labeled.

3) Labels

a) Labels on incoming containers must not be removed or defaced.
b) The date a chemical is received will be tracked via the bar code affixed to the container.
c) Containers into which a chemical is transferred must be labeled with an identifier (such as a chemical name or trade name) as it appears on the MSDS.

4) Storage Of Chemicals

a) Never store reagents on the floor.

b) Provide trays under large containers of chemicals to contain leaks.

c) Chemicals will be segregated into the following classes and stored in proper cabinets:

INORGANICS (I)

I1 Metals, hydrides (store away from water and reducing agents. Flammable solids are stored in the flammable cabinet).

I2 Halides, iodides, sulfates, sulfites, thiosulfates, phosphates, halogens.

I3 Nitrates, nitrites, azides (NOT AMMONIUM NITRATE).

I4 Hydroxides, oxides, silicates, carbonates, carbon.

I5 Sulfides, selenides, phoshides, carbides, nitrides.

I6 Bromates, chlorates, perchlorates, chlorites, perchloric acid, peroxides, hypochlorites, hydrogen peroxide.

I7 Arsenates, cyanides, cyanates.

I8 Borates, chromate's, manganates, permanganates.

I9 Acids, mineral and inorganic.

<u>ORGANICS (O)</u>

O1 Acids, anhydrides, peracids.

O2 Alcohols, glycols, amines, amides, imines, imides.

O3 Hydrocarbons, esters, aldehydes.

O4 Ethers, ketones, ketenes, halogenated hydrocarbons.

O5 Epoxy compounds, isocyanates.

O6 Peroxides, hydroperoxides, azides.

O7 Sulfides, polysulfides, sulfoxides, nitriles.

O8 Phenols, cresols.

The above table should be consulted in determining the proper classification of chemicals. If uncertain into which category a chemical may fall, consult the MSDS or other reference source.

O2, O3 and O4 chemicals must be stored in a flammable storage cabinet or a storage room. I9 and some O1 chemicals are to be stored in the acid storage cabinets.

d) A chemical inventory shall be maintained for all chemicals in the laboratory. On an annual basis the chemicals in the inventory will be reviewed, and at that time a decision will be made whether to keep or dispose of a chemical. This decision will be based on:

- The self-life of the material
- Whether the liquid has become cloudy
- Whether the chemical has changed color
- Whether the material has become caked
- Whether there are solids in a liquid or liquid in a solid
- Evidence of a reaction with water

- Damage to the container
- Formation of crystals

Any of these changes may have created a hazardous situation. The ES&H Group will be consulted for handling and disposal information.

5) Air Monitoring

Air monitoring will be performed whenever there is reason to believe exposure levels of a substance exceeds the action level (or in the absence of an action level, the permissible exposure limit). Air monitoring will be performed by the TD ES&H Group.

Employee exposure monitoring shall be performed for the following:

a) To determine employee exposure to a confirmed or suspected carcinogen as defined by OSHA or ACGIH.

b) If, in the judgment of the TD ES&H Group, routine exposure could potentially expose employees above the OSHA action level or to 1/2 of the PEL or TLV, where there is no applicable OSHA PEL.

c) Upon request of an employee or the CHO.

d) As required by the OSHA regulations.

e) If an employee reports signs and symptoms as a result of exposure.

If the exposure exceeds 1/2 of the TLV, the TD ES&H Group will issue personal protective equipment to the employee until engineering controls reduce the exposure.

6) Ventilation

a) Annual Survey

The ES&H Group will conduct an annual ventilation survey to measure the average velocity of all ventilation units in the MDTL. This is a requirement of the Fermilab ES&H Manual Chapter 5091, Hazard Control Ventilation.

b) Modifications

Ventilation units shall not be modified unless it has been determined that the modification will not impair worker protection.

7) Fume Hoods

a) The hood should be used when working with toxic chemicals.

b) The sash should be used at the marked height. This assures an average face velocity of 100 feet per minute. The average face velocity is checked annually by the ES&H Group.

c) Hoods shall not be used as a means for disposing or long term storage of chemicals.

d) Check the operation of the hood before each use.

e) Users must keep their face outside the plane of the hood sash.

f) Equipment should be placed as far back in the hood as possible. All activities should be carried out at least 6 inches back from the front edge of the hood.

g) When the hood is not in use, turn off the fan and close the sash.

h) If chemicals must be stored in the hood, keep the fan on.

8) Abrasive Blasting Cabinet

a) An abrasive blasting cabinet is available for use in the MDTL. The blasting cabinet must be inspected by the operator before each use.

b) The abrasive blasting cabinet is checked annually for airflow by the ES&H Group during the annual ventilation survey.

9) Eye Wash and Deluge Shower

A plumbed eyewash and deluge shower are provided in each laboratory. These are tested weekly and the testing is documented. Employees are instructed in the proper use of this equipment by the CHO.

If a chemical splash to the eyes occurs, the employee will flush the eye for at least 15 minutes. In the meantime, someone else in the area must dial 3131 to summon medical assistance. The individual may be taken to the Medical Department in those instances of minor injury.

10) Housekeeping

Good housekeeping practices in the laboratory contribute greatly toward a safe work environment. A clean work area is much safer than a cluttered or dirty one. Here are a few good housekeeping helpful hints:

- Keep all aisles, hallways, and stairs clear of all chemicals and other obstructions;
- Keep all work areas, especially workbenches and desks clear of clutter and obstructions;
- All working surfaces and floors should be cleaned regularly;

- Access to emergency equipment, drench showers, eyewashes and exits shall not be blocked;
- Return chemicals to the proper storage areas at the end of each workday;
- Label all chemical containers with their identity;
- Promptly clean up all chemical spills and dispose of properly;
- Do not store chemicals in aisles, stairways, on workbenches or desks, on floors or in hallways;
- Hazardous wastes shall be kept in the proper containers and labeled;
- Do not dispose hazardous wastes in the regular trash.

11) <u>Inspections</u>

Formal safety inspections are performed on a quarterly basis by trained internal inspectors. All deficiencies are entered as findings into the Laboratory's ESHTRK Database.

12) <u>Medical Program</u>

Medical examinations, through the Fermilab Medical Department, are available on a biannual basis. Additional medical examinations are available for the following conditions:

a) An employee develops signs and symptoms associated with a hazardous chemical to which the employee may have been exposed in the laboratory.

b) Exposure monitoring reveals an exposure level routinely above the OSHA action level or 1/2 the PEL or TLV, where there is no PEL.

c) An event such as a spill, leak, explosion, or other occurrence results in the likelihood of hazardous exposure.

The treating physician will be provided with the identity of the hazardous chemical(s) to which the employee may have been exposed and a description of the conditions under which exposure occurred.

13) <u>Personal Protective Equipment</u>

The following personal protective equipment (PPE) is available:

- Chemical Splash Goggles
- Gloves (Various Types)
- Face Shield

- Rubber Aprons
- Safety Glasses

The use of PPE in the MDTL and SRF Materials Laboratory shall be determined by the corresponding CHO.

Safety Glasses shall be worn at all times in the Laboratory. Wearing contact lenses under safety glasses is acceptable except in dusty conditions.

PPE shall be kept in a clean and sanitary condition. PPE shall be stored in Cabinet # 13. PPE is periodically inspected by the CHO and defective equipment replaced.

a) Gloves

i) Gloves of the appropriate material and size shall be worn when there is the possibility of contact with a toxic or hazardous material. The CHO in consultation with the ES&H Group will select the proper glove with the required degree of protection for the substance(s) being handled.

ii) Gloves shall be inspected for tears or punctures and tested before each use. Each glove should be tested by inflating the air, tie off the wrist area and then submerge the glove into a container of standing water. If any air bubbles are detected, dispose the gloves. Even new gloves should be inspected and tested before each use.

iii) The cuff of the glove should fit up under the cuff of the lab coat.

iv) After handling a toxic or hazardous material with gloves, wash the glove before removal.

v) Periodically replace gloves. If corrosive or irritating chemicals are used, gloves shall be inspected frequently. They should be rinsed with water and dried thoroughly during lengthy processes. If they have been splashed with a chemical or you suspect they have been, they should be rinsed with water and thoroughly dried.

vi) The following are examples of some glove selections. These are only suggestions. Adequate substitutes may be used, but it is extremely important that a glove is selected based on its known protection against the chemical agent. Consult with the TD SSO with any questions.

1. Broken glass -- leather
2. Acetone -- butyl rubber
3. Benzene -- polyvinyl alcohol, viton
4. Chloroform -- polyvinyl alcohol, viton
5. Formaldehyde -- butyl rubber, nitrile rubber, polyethylene
6. Hydrochloric Acid -- Butyl rubber
7. Methylene Chloride -- polyvinyl alcohol

8. Phenol -- butyl alcohol
9. Toluene -- Polyvinyl alcohol, Teflon, viton
10. Hydrofluoric acid -- butyl rubber
11. Phosphoric acid -- butyl rubber

b) Clothing

i) No shorts can be worn when working with chemicals.

ii) If there is a possibility of contact with a chemical, a lab coat shall be worn. If corrosive or irritating chemicals are used, a lab apron shall be worn under a lab coat.

iii) All jewelry and watches should be removed if corrosive or irritating chemicals are being used.

c) Shoes

Safety shoes shall be worn in the laboratory when there is a hazard that could cause a foot injury. Wearing any type of open-toed footwear in the laboratory is strictly prohibited.

d) Respiratory Protection

The ES&H Group will be consulted on the need for and the selection of respirators.

e) Eye Protection

i) Eye protection shall be worn at all times in the laboratory.

ii) Contact lenses are prohibited in a dusty environment.

iii) When handling potential eye hazards, more protection shall be required. Chemical splash goggles must be worn when working with strong acids, bases, and hydrofluoric acid.

1. Chemical Splash Goggles

   These are specifically designed to protect the eyes in the event of a chemical splash. The side ventilation slots in normal goggles are eliminated or protected.

2. Face Shields

   These must be used when the face and neck need protection in the event of a chemical splash from any caustic material or carcinogenic material. A face shield <u>must</u> be worn in conjunction with chemical splash goggles.

14) Records

a) Injury/Illnesses

Injury/illness investigations are retained in the Computerized Accident Illness Recording System (CAIRS) database.

b) Chemical Hygiene Program

Each CHO is responsible for maintaining all documentation concerning the Chemical Hygiene Program.

15) Accidents

a) All accidents must be reported immediately to the supervisor.

b) Eye contact: If a chemical splash to the eyes occurs, the employee must promptly flush the eyes for 15 minutes. Another employee must summon medical attention by calling 3131.

c) Ingestion: If the employee ingests a chemical call 3131.

d) Skin Contact: If a chemical contacts the skin, the affected area must be flushed with water and any contaminated clothing removed. If the symptoms persist after washing, the employee must be taken to Medical.

16) Spills

This applies to small spills (less that 1 liter of liquid). Large spills will be handled by the Fire Department (Call 3131).

a) Liquid Spills

i) Caustic

1. Wear a face shield, rubber gloves, and rubber apron.

2. Use the spill control pillows to confine the spill if necessary.

3. Dilute the spill with an equal amount of water.

4. Sprinkle Alkali Neutralizer (Spill Pac 1) on spill.

5. Add water as required to control heat evolution.

6. When neutralization is complete, there will be a color change from blue to pink.

7. Once the spill is neutralized, use Spill Pac 2 to absorb the spill.

8. Scoop material into a plastic bag.

9. Contact the TD Waste Coordinator for disposal.

ii) Flammable Solvents

1. Eliminate all sources of ignition.

2. Wear a face shield, impervious gloves (neoprene), and rubber apron.

3. Use spill control pillows to contain the spill.

4. Sprinkle flammable solvent adsorbent (spill kit) on the spill.

5. Scoop dry mixture into a plastic bag.

6. Contact the TD Waste Coordinator for disposal.

iii) Acids

1. Contain the spill with spill control pillows. DO NOT USE FOR A HYDROFLUORIC ACID SPILL.

iv) Mercury

1. Contact the ES&H Group in the event of a mercury spill.

17) <u>Training and Information</u>

a) All MDTL employees, SRF Materials Laboratory employees, and authorized users will receive initial safety training from the CHO, and will receive refresher training when new hazards are introduced into the laboratory. Additional training will be given whenever a new process or particularly hazardous chemical is introduced. All training provided by the CHO will be documented in TRAIN.

b) Employee training will cover:

i) The contents and availability of the OSHA Laboratory Standard 29 CFR 1910.1450.

ii) The contents and availability of the Chemical Hygiene Plan.

iii) The hazards of the chemicals used by employees in the laboratory.

iv) The Permissible Exposure Limit (PEL) for OSHA regulated substances or recommended exposure limits (TLVs) where there is no applicable OSHA PEL.

v) The signs and symptoms associated with exposure to hazardous chemicals used in the laboratory.

vi) The location and availability of reference materials on chemical hazards, and the safe handling, storage, and disposal of hazardous chemicals.

vii) The measures employees can take to protect themselves from chemical hazards such as appropriate work practices, emergency procedures, and personal protective equipment.

viii) Special precautions needed for carcinogens and especially hazardous chemicals.

18) Material Safety Data Sheets (MSDS)

A material safety data sheet (MSDS) is available on-line at the ES&H Section web site for each chemical in the laboratory:

http://www-esh.fnal.gov/pls/default/msds_search.html

A MSDS must be requested for each new chemical that is ordered. The CHO shall assure that a MSDS is received and forwarded to the TD ES&H Group for inclusion in the database. If a MSDS is not received, the manufacturer will be contacted and a MSDS requested. A MSDS must be attached to the Purchase or Procard Requisition for any new chemical before proceeding to ES&H review.

19) Waste Disposal

The MDTL and SRF Materials Laboratory comply with the Fermilab ES&H Manual Chapter 8021 on the disposal of chemical wastes. FESHM 8021 can be viewed at:

http://www-esh.fnal.gov/FESHM/8000/8021.pdf
http://www-esh.fnal.gov/FESHM/8000/8021.doc

D. STANDARD OPERATING PROCEDURES

1) General Rules

a) Eye protection must be worn at all times in the laboratory. Depending on the severity of the hazard, chemical goggles and/or a face shield may also be required.

b) Identify where potential sources of danger or hazards are located. Read all posted signs. Identify the locations of eyewash stations, emergency showers, the Chemical Hygiene Plan, and access to MSDSs. If hydrofluoric acid is used, identify the location of the antidote.

c) All equipment shall be inspected before each use. Defective equipment shall be removed from service until repaired.

d) Good housekeeping shall be practiced at all times. Keep floors, shelves, and tables free from unnecessary apparatus and materials. Aisles, doorways, and exits shall not be blocked. Apparatus not in use shall be stored in a cabinet. Set-ups must be set back from the edge of the bench. Clean up the laboratory before leaving at the end of the day.

e) Broken glassware shall be placed in a special container.

f) Horseplay, pranks, or other acts of mischief are dangerous and prohibited.

g) Never pipette or start suction by mouth.

h) Safety shoes shall be worn in the laboratory when there is a hazard that could cause a foot injury. Wearing open-toed footwear in the laboratory is prohibited.

i) Confine loose hair and clothing at all times.

j) Consumption of food or beverages in the laboratory is prohibited.

k) Food and beverages shall be stored in refrigerators for food only.

l) There shall be no unauthorized experiments. All experiments must be approved by the CHO.

m) Work with new chemicals only after reviewing the MSDS.

n) Never perform experimental work alone.

2) <u>Carcinogens</u>

a) The Materials Development and Testing Laboratory uses the following confirmed or suspect carcinogens as defined by OSHA or ACGIH:

APPENDIX A CHEMICALS

Methylene Chloride
Benzene
Carbon Tetrachloride
Chloroform
Formaldehyde
Nickel
Cadmium
Methylene Dianiline (in Tonox and Epon Curing Agent Z)
1,4 Dioxane

b) The use of these materials will be kept at a minimum. Whenever possible, a less toxic substance will be substituted.

c) The Medical Department is informed of all employees potentially exposed to these substances. This notification is updated annually.

d) An inventory of carcinogens used in the Material Development Laboratory is updated annually. The inventory contains:

- Name of the carcinogen
- Quantity of the carcinogen on hand
- Location the carcinogen is stored

e) Containers of carcinogenic chemicals are labeled according to the Fermilab ES&H Manual Chapter on Hazard Communication. In addition the words "CANCER HAZARD" must appear on the label.

f) The ES&H Group must approve any use of carcinogens. The ES&H Group will provide personnel air monitoring for employees using the material. If the employee exposure exceeds the permissible exposure limit, a Regulated Area will be established.

g) The following personal protective equipment (at a minimum) shall be used:

- Laboratory coat
- Impervious gloves (refer to SOP on gloves)
- Face shield

If the personal protective equipment is contaminated, it must be placed in a zip lock bag and disposed according to the Fermilab ES&H Manual Chapter 8021.

If the material gets into the employees eyes or on the skin, flush the area with water and the employee will be taken to Medical.

h) All carcinogens must be used under a laboratory hood.

APPENDIX A

CHEMICAL HYGIENE PROGRAM
TRAINING OUTLINE

3/05

I SAFE WORK PRACTICES

A) Rules and Responsibilities

1. Fermilab ES&H Manual
2. Chemical Hygiene Plan
3. OSHA and DOE

B) General Conduct

1. Perform only authorized experiments and operations
2. Practice Good Housekeeping
3. No "horseplay"
4. Follow safety rules
5. Wear safety glasses and shoes when in the lab
6. No food, drinks, or smoking in the lab

C) Handling Equipment and Chemicals

1. Inspect all equipment before each use. If it is defective, notify the CHO.
2. Use toxic chemicals under the hood
3. Storage of chemicals
4. Waste disposal
5. Shield vacuum equipment

II. TYPES OF HAZARDOUS MATERIALS

A) Physical Hazards

1. Fire Hazards

    a. Pyrophorics
    b. Flammables
    c. Combustibles
    d. Oxidizers

2. Health Hazards

    a. Chemicals

        i. Organic Solvents
        ii. Corrosives
        iii. Sensitizers
        iv. Irritants
        v. Carcinogens

3. Routes of Entry

    a. Ingestion
    b. Inhalation
    c. Skin Contact

4. Material Safety Data Sheets

5. Chemical Labeling

III. PERSONAL PROTECTION

A) Eye and Face Protection

1. Safety Glasses
2. Chemical Splash Goggles
3. Face Shield

B) Hands, Arms, Foot and Body Protection

1. Gloves
2. Safety Shoes
3. Aprons, and Lab Coats

C) Respirators

1. Ventilation should be adequate so respirators should not be necessary

IV. Emergency Procedures

A) Review of emergency procedures

1. Dial 3131
2. Tornado
3. Fire
4. ODH
5. Accident

B) Emergency eye wash and shower

APPENDIX 8

P. Bauer / Y. Terechkine – Fermilab/TD
08/19/2004
Memo regarding external pumping station for IB3-SRF Materials Lab

In the frame of recent efforts at Fermilab to develop expertise, infrastructure and a technology base for SRF technology, a new SRF materials lab was recently created. One of the systems that need to be installed in this new lab space is a test station for the measurement of material properties at the low operating temperatures of SRF cavities. Such a facility consists of a liquid helium cryostat, into which samples of the materials used for SRF applications are inserted and electrical or thermal measurements performed. Since SRF cavities typically operate at temperatures below 2 K, i.e. below the boiling point of helium in atmospheric conditions, an additional pumping capacity is needed to reduce the vapor pressure on the helium bath to the mbar range and thus lower the temperature to the specified level. Such a pumping system cannot be installed inside the laboratory because of noise, vibration and heating issues. We therefore propose that such system be installed at the outside of the building, with a pumping port connecting it to the test station inside the laboratory space. This outside space would be the eastern front of the IB3 building.

The pumping system required for the SRF lab facility is very similar to a system currently operating in the Engineering Lab on the groundfloor of ICB. A photo of this system taken recently is therefore shown below. The system consists of two pumps, a roots blower with a large pumping capacity to pump in the low-pressure regime and a more standard rotary vane pump for pumping from atmospheric conditions to 1 Torr. The total dimensions of the system are 24"x 24"x 9". The pumps need to be encased. A pumping line of diameter ~3" connects it to the test station inside the laboratory space. It is an air-cooled system with ~10 kW worst-case heat rejection (but low operating duty cycle). The vibrations generated by such a system are considerable. Given the experience with the existing systems we do not expect any particular technical difficulties related to the installation or operation of such a system.

Noise, vibration and heating warrant that such a pumping station be installed at the outside, enclosed in reasonably soundproof housing. The area in which we intend to place the pumping station along the eastern flank of the current IB3 building is currently being envisioned as the future atrium area connecting the IB3 building to the future IB3 extension, in the planning stages today. Especially due to noise issues the pumps cannot remain within the envisioned atrium area. Therefore two possible solutions for the future of the SRF materials lab beyond the start of the IB3 extension construction were investigated:

a) keeping the SRF materials lab where it is and placing the pumps on the roof of the IB3 building or in a different location outside IB3 (e.g. outside the north-wall of the building), or
b) moving the entire lab to a different location (possibly the IB3 extension);

Ad a) The major disadvantage of moving the pumps to a remote location is the additional cost for the long pumping lines and for the systems needed to mitigate vibration effects if the pumps are placed on the roof. Also, a remote control system of the pumps needs to be installed and maintenance is a little bit more tedious. The issue of an increased pump-down time as a result of extended piping between the Dewar and the pumps was also investigated. Simulation of the pump-down process by T. Peterson, assuming a 2” ID, 10 m long pumping line indicate that the pump-down time remains reasonable (~ 2 hrs) and therefore this issue is considered to be less severe than initially thought.

Ad b) Moving the entire SCRF materials lab to a different location (possibly the IB3 extension) also involves cost and lost operating time due to the moving operation. Furthermore, available lab space is not yet defined. The possibility of moving the SCRF materials lab to the IB3 extension was also discussed, but no such possibility is foreseen now in the (fairly mature) occupancy plans for the IB3 extension.

Summarizing the above said we believe that keeping the SRF lab in the current location and moving the pumping station to a different place once the IB3 construction has started is probably the cheapest and least disruptive solution. The pumping station can be moved to either the north side of the IB3 building or the roof. A ~4” diameter pumping line would be needed to provide sufficient conductance.

APPENDIX 9

CALCULATION OF THE BASIC PARAMETERS FOR THE SRF MATERIALS LAB PUMPING SYSTEM

P. Bauer, C. Boffo, R. Rabehl, T. Peterson
Mar. 20, 2005

BASIC PRINCIPLE

Pump on liquid helium bath (max liquid volume 100 lit) until vapor pressure drops to ~ 7 mbar to cool bath to 1.6 K. Pump schematic is shown below:

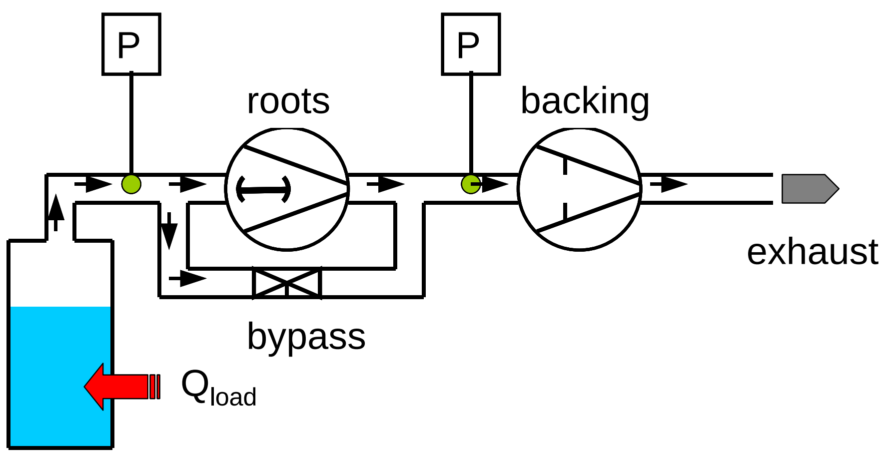

Figure 1: Pump schematic for helium cooling process;

ESTIMATE OF REQUIRED PUMPING SPEED DURING COOL-DOWN PROCESS

To cool a 100 lit liquid helium bath by ~1 K per hour (and that thus sets a cooling time) the following amount of cooling power needs to be provided approximately:

$$Q_{cool} = V \times \rho \times C_p \times \frac{1K}{3.6 \cdot 10^3 s} \approx 100 lit \times \frac{150 g}{lit} \times 5 \frac{J}{g-K} \times \frac{1K}{3.6 \cdot 10^3 s} = 21W$$

A similar cryogenic system has been estimated to have a static heat load to the liquid helium of 7 W. The total power load for the liquid bath during cool-down can therefore be estimated to be 28 W.

The latent heat of vaporization of helium is 20 J/g. At 28 W power we have to expect a total mass-flow of:

$$\dot{m} = \frac{Q_{tot}}{\Delta H} = \frac{28W}{20\ {}^{J}\!/_{g}} = 1.4\frac{g}{s}$$

This mass flow rate certainly overestimates the actual flow rate in the system, especially once the bath is at the desired low temperature and only the evaporation due to the static heat load occurs. On the other hand, the calculation does not take into account the effect of continuous filling of the Dewar, which would introduce an additional gas-load even after the desired bath temperature was reached.

The pumping station includes two pumps in series: a vane backing pump (P1) that takes care of the high pressure drop between atmosphere (~1013 mbar) and 50 mbar, and a roots-blower (P2) working on the low pressure side allowing to reach pressures below 50 mbar. This setting is achieved by placing a bypass around the roots-blower (P2) that restricts the use of this pump only for inlet pressures below 50 mbar. This bypass sets also the minimum inlet pressure of the vane backing pump (P1) defining its required maximum pumping speed.
In the following calculations it is assumed:

- The target helium bath temperature is 1.8K corresponding to a reference pressure of 16 mbar.
- The pressure loss in the piping between the Dewar and the pumping station is neglected.
- The pumps receive the helium gas at room temperature and at variable pressures requiring a heat exchanger described in Appendix 8.

The pumping speed (V) required for the two pumps is calculated at the minimum inlet pressure: 16 mbar for P2 and 50 mbar for P1. The ideal gas law is used to calculate the helium density at the two pressures:

$$\rho = p\frac{m}{RT} = p(mbar)\frac{4\dfrac{g}{mole}}{83.14\dfrac{mbar-lit}{mole-K}\times 300K} \quad \left(\frac{kg}{m^3}\right)$$

◎ = 0.0080 kg/m³ @ 50 mbar 300K P1
◎ = 0.0026 kg/m³ @ 16 mbar 300K P2

$$V = \frac{\dot{m}}{\rho(P)} \quad \left(\frac{lit}{\sec}\right)$$

Given the 1.4 g/s gas flow, the pumping speed V at 50 mbar is 175 lit/s, corresponding to 371 cfm, and 545 lit/s corresponding to 1155 cfm at 16 mbar.

The combined pump system in a recent quote (I, Leybold) is specified to provide 1140 cfm at 0.1 mbar. Another quote (II, Pfeiffer) is even better: 1343 cfm at 1 mbar.
However the pump system consists of two pumps, a blower and a backing pump. Due to the automatic by-pass, the backing pump is working both during start up at atmospheric pressure

and during cool-down while the roots-blower is kicking in at the low-pressure end. Therefore, the specific pumping speed restrictions of each pump apply during the different phases of system operation. Together with the static heat load in the Dewar (and the related volumetric flow rate) these parameters are mostly indicative of the minimum temperature than can be achieved in the Dewar. The cool-down phase (and therefore the cool-down time), is limited by the backing pump pumping speed which is typically ~500-600 cfm in the systems quoted above, while the minimum reachable pressure is limited by the root blower performance. Since the backing pump works for longer periods of time its motor needs to be large enough to sustain this work for extended durations. The pumping power requirements are discussed next.

CALCULATION OF PUMP POWER CONSUMPTION

In order to correctly design the pumps, one has to calculate the thermodynamic work performed on the helium during the compression. The ideal gas properties are considered for these calculations. Ideally the most efficient way to compress a gas is an isothermal transformation where the fluid is compressed at constant temperature with low work. In reality a volumetric machine like the vanes and the root vacuum pumps we are dealing with, perform a polytropic (isentropic) transformation. Since the machine is not ideal the irreversibility of the process must be taken into account as well. Results of the isothermal, reversible and irreversible polytropic transformations will be compared during the calculations.

The isothermal power consumption is calculated as follows:

$$P = \dot{m}\frac{R}{M}T_{in}\ln\left(\frac{p_{out}}{p_{in}}\right)\ (W)$$

The isentropic power consumption is calculated as follows:

$$P = \dot{m}\frac{m}{m-1}\frac{R}{M}T_{in}\left[\left(\frac{p_{out}}{p_{in}}\right)^{\frac{m-1}{m}}-1\right]\ (W)$$

Taking into account the irreversibility:

$$P = \dot{m}C_pT_{in}\left[\left(\frac{p_{out}}{p_{in}}\right)^{\frac{m-1}{m}}-1\right]\frac{1}{\eta}\ (W)$$

Figure and Figure 3 show the thermodynamic power consumption for each pump as a function of input pressure. The needed power in order to reach 1.8K with a 1K/min cool down rate is 6kW for P1 and 1.5kW for P2. Given the continuous gas-load from the helium bath, the motor power needs to be chosen carefully. As discussed in the next section existing systems at Fermilab use large pump motors to cool down Dewars.

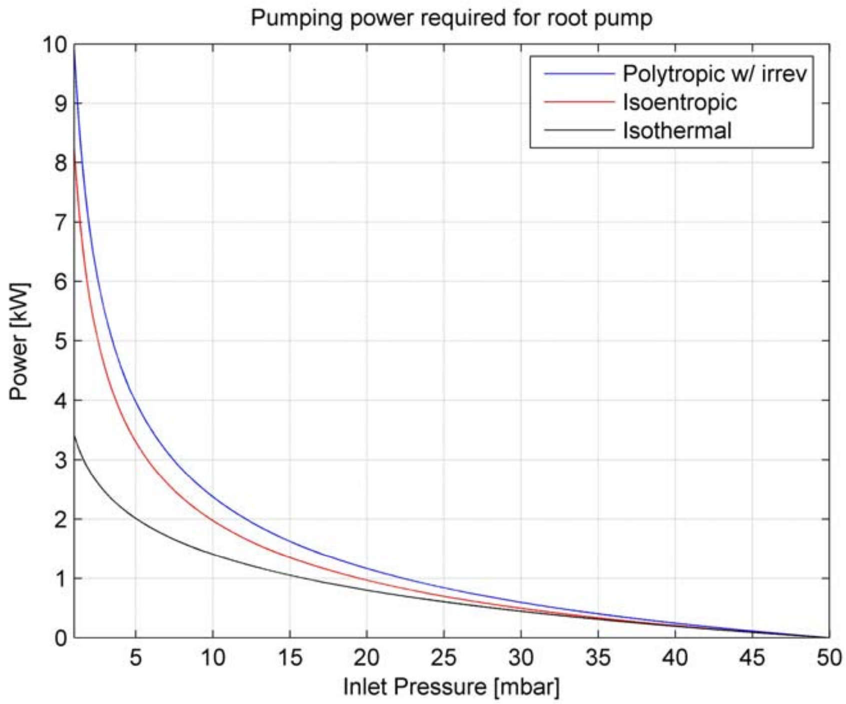

Figure 2: Calculation of low pressure pump power consumption between 5 and 50 mbar pressure differential across the pump for a given 1.4 g/s helium flow-rate at room temperature and outlet pressure of 50 mbar.

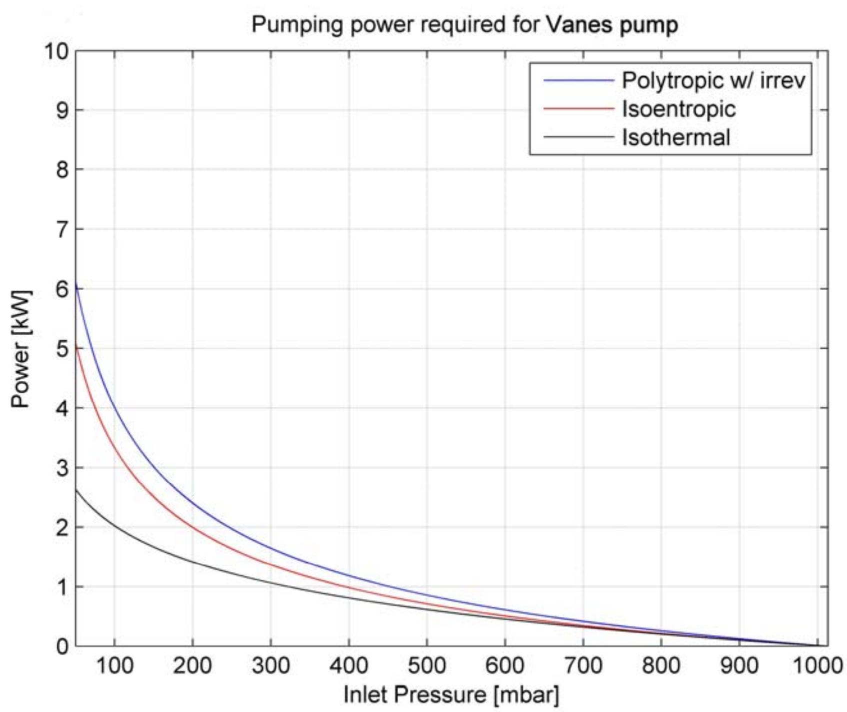

Figure 3: Calculation of roughening pump power consumption between 50 and 1000 mbar pressure differential across the pump for a given 1.4 g/s helium flow-rate at room temperature and outlet pressure of 1013 mbar.

COMPARING DIFFERENT PUMP SYSTEMS

The following table compares different pump systems at Fermilab, which are used to cool liquid helium to <2 K. The comparison shows that the systems quoted for the SRF materials lab use a blower motor power that is weaker by ~2 than the one used for the new MTF. It is, however, a given, that the heat exchanger between the Dewar and the blower will introduce a conductance limitation that will reduce the actual mass flow in the SRF materials lab system. A similar argument applies to the case of the backing pump motors, which are also larger in MTF-new.

Table 1: Comparison table.* nominal flow indicated – but flow is restricted through thin diameter pumping line.

System	Dewar	Vol flow	Vol flow	mdot	Blower	Rough
	(lit)	(cfm)	(m^3/hr)	(g/s)@16mbar	kW	kW
MTF old	-	-	-	2.5	40	100
MTF new	-	1225	2080	1.5	20	60
Eng Lab	100	589*	1000*	0.7*	5	10
SRF lab quote I	100	1140	1937	1.4	7.7	18.7
SRF lab quote II	100	1343	2282	1.6	7.7	18.5
calculated	100	1155	1962	1.4	1.5	6

CALCULATING THE PUMP-DOWN TIME SYSTEMS

The plot below shows the time for pump-down to 1.74 K for 100 liters of helium with the Leybold vacuum pumping system. Pump-down takes a little under 2 hours, with an ultimate temperature of about 1.7 K. (Assuming a steady heat load of 7 Watts into the bath.) A 6-inch nominal size pipe 15 meters long results in a negligible pressure drop to the pumps. The model assumes a mass flow rate, which is calculated from the specified pumping speed in quote I (550 cfm @ 0.1 Torr, which is scaled to 0.7 g/s at 16 mbar via the linear pressure dependence of the density). The model then iteratively calculates the bath temperature as a function of time, taking into account:

- the variation of bath saturation vapor pressure (and therefore pressure in the pumping line as well as pumping speed) with bath temperature;
- the variation of vapor enthalpy with bath temperature;
- the variation of evaporated mdot as a function of pumping speed.

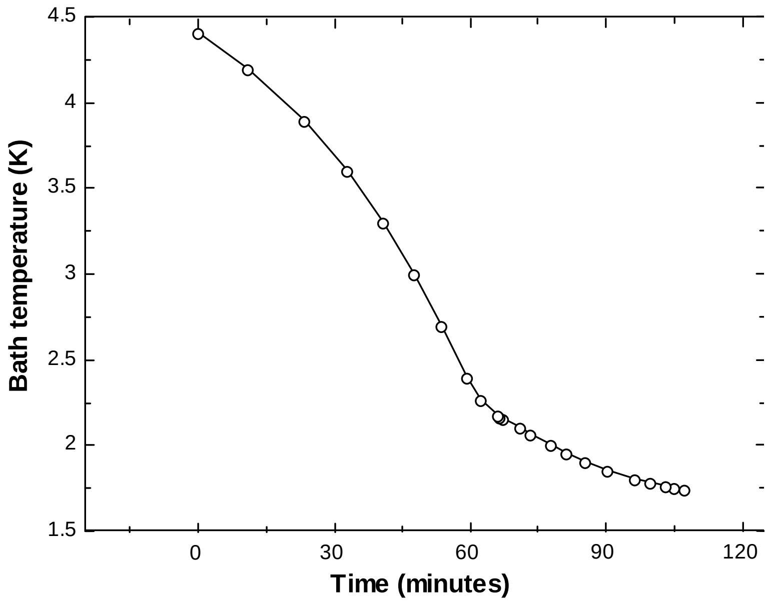

Figure 3: Cool-down profile calculated for a 0.7 g/s flow rate (550 cfm).

APPENDIX 10

CALCULATION OF THE BASIC PARAMETERS FOR THE SRF MATERIALS LAB PUMPING SYSTEM HEAT EXCHANGER

C. Boffo

Oct. 13, 2005

BASIC PRINCIPLE

The pumping system for the cryogenic setup of the lab requires the helium at its inlet port to be at room temperature. Primarily the pumps will be used to reach low pressure in the cryogenic vessel in order to operate with liquid helium down to 1.8 K or to generate a controlled atmosphere of helium gas at temperatures between 4.2 K and 300 K. For the purpose, in order to assure that the fluid reaching the pumps is at the proper temperature, a heat exchanger is needed. For equipment safety a temperature sensor at the inlet of the pump system switches the pumps off when the reading reaches a value below a fixed limit. Moreover a bypass allows operating with the heat exchanger disconnected when the flow rate is small or if the pressure drop in the lines becomes a considerable factor. The schematic of the system is shown in Figure 1.

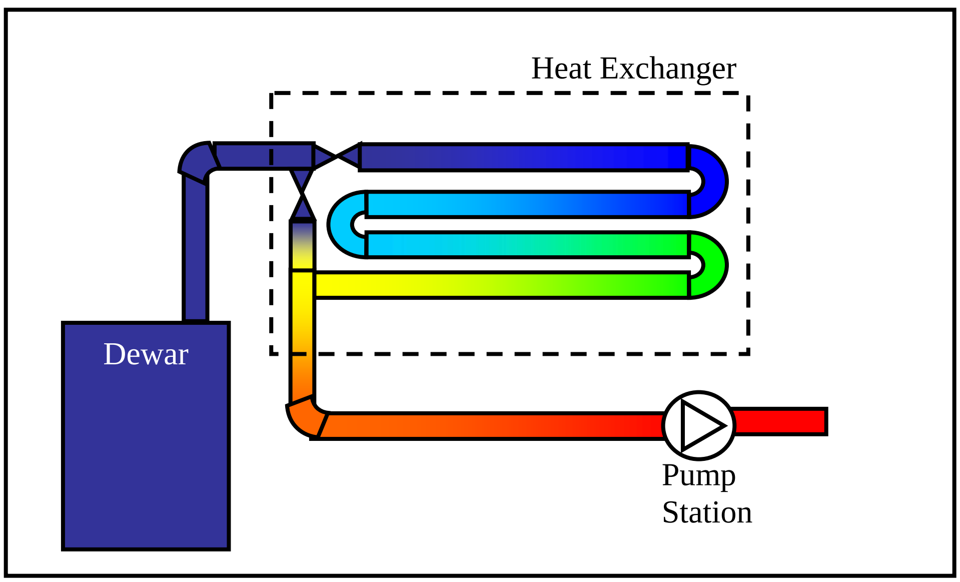

Figure 1: Schematic of the heat exchanger;

HEAT EXCHANGER DESIGN

The simplest design for this heat exchanger is a tube exposed to room temperature air in natural convection. To be practical the tube must be bent to generate several parallel passes in order to reduce the overall length and the foot print. Moreover it will be placed in the same shelter as the pumps in order to avoid water condensation on the floor of the lab due to frosting of the piping. Second order advantages gained by placing the heat exchanger in the shelter are: the reduction of noise in the lab due to high gas speed in the piping and the effect of higher room temperature in the shelter due to the equipment operations. In order to improve the heat exchange in future will be possible to add resistors on the pipe allowing for higher temperature differential and a fan to optimize the air side heat exchange coefficient. As shown later the last option is not very effective since the heat flow during the process is mainly regulated by the helium thermal properties at low pressure.

PROBLEM MODEL

The pressure during the heat exchange is regulated by the pumping system and since the pressure drop in the pipe is negligible, all the process can be considered isobaric. Given the specifications of the system, the model is evaluated at 16 mbar assuming a helium mass flow rate of 1.4 g/s.

The temperature range of the process, given the constant cross section of the tube, results in a strong change of density in the gas with equivalent rise of the gas speed. For this reason the dimension of the tube is calculated considering the flow rate at the pump inlet at room temperature. It is clear that the temperature together with the speed differentials during the process have a significant impact on the thermal and fluidynamic properties of helium. The helium velocity along the pipe for different cross sections is shown in Figure 2.

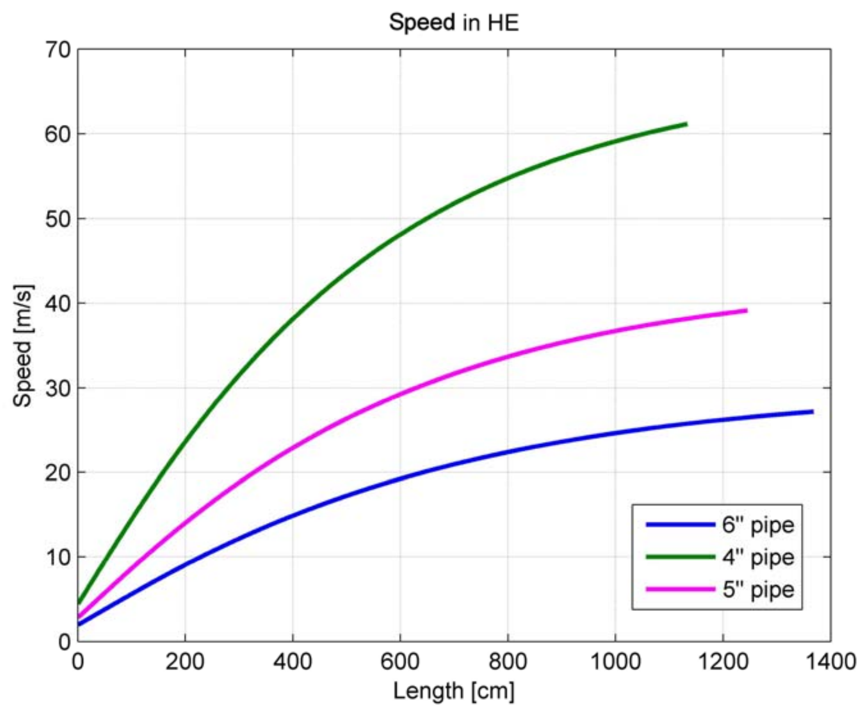

Figure 2: Helium speed for a 1.4 g/s mass flow rate at 16 mbar with temperature rising from 10 K to ~273 K along a pipe of 4", 5" or 6" diameter. The different length relates to the different heat exchange average coefficients related to each pipe diameter;

The choice of the pipe diameter is a free parameter in the attempt to maximize the global heat exchange coefficient while keeping the top helium speed and the pressure losses within reasonable values. Diameters between 4" and 6" look suitable to the purpose (see Figure 2 and calculations below).

In order to rise the temperature of the given helium mass flow from 10 K to 300 K one needs to exchange a heat Q of:

$$Q = \dot{m} C_p \Delta T \quad (W) \qquad [1]$$

equal to ~2 kW assuming an average specific heat coefficient of 5.2 J/KgK.
The warm up of the helium involves different heat exchange contributions:

- Convection and conduction within the helium
- Convection between helium and the pipe internal surface
- Conduction along the pipe
- Convection between the pipe external surface and air

Since the properties of helium and its speed change significantly, it is not possible to design the heat exchanger using standard ◎-NTU or ◎Tml methods. The local global heat exchange coefficient must be calculated at each point of the tube. The heat exchange per unit length of tube is:

$$Q_{HE} = KA\Delta T \quad (W/m) \qquad [2]$$

Where KA is the product of global heat exchange coefficient and exchange reference surface.

$$KA = \frac{1}{\frac{1}{KA_1} + \frac{1}{KA_2} + \frac{1}{KA_3}} = \frac{1}{\frac{1}{\alpha_i A_i} + \frac{\ln\left(A_e/A_i\right)}{2\pi l\lambda} + \frac{1}{\alpha_e A_e}} \qquad [3]$$

The three elements in KA are the convection contribution for the helium side KA_1, the conduction through the pipe thickness KA_2 and the convection between pipe external wall and air KA_3. Out of the three, the conduction in the pipe is negligible being orders of magnitude higher than the other two. Between the two other components the most limiting is the helium side even considering air in natural convection. In general on the air side, for the following calculations, a fixed film coefficient of 20 W/m^2K is assumed.
The calculation have been performed initially in MatLab and later using a one dimensional model in ANSYS in order to take into account the conduction along the tube. An additional analysis was performed in FEMLAB in order to understand the effect on air temperature due to the stacking of multiple horizontal pipes to save foot print. The helium properties where evaluated at 16 mbar using HEPAK.

MATLAB SOLUTION

The basic concept behind this calculation is to divide the pipe into small length elements and, for each of them (starting form the cold side at 10 K), balance the heat adsorbed by the helium with the heat exchanged with the outside world. In terms of equations this means that Q equals Q_{HE}*L in equations 1 and 2. Given the inlet temperature, the result is the temperature of helium at the outlet of the small part. This number is then the input for the next element. The process is over when the helium temperature reaches 273 K. the helium properties are evaluated at the local temperature using a table imported from HEPAK. The film coefficient for the helium side is defined at each step by recalculating Reynolds, Prantl and Nusselt numbers given the fluid properties and the recalculated speed. As mentioned before, the results of this method do not take into account the conduction along the tube which results in a longer length. Figure 4 shows the results for different pipe diameters. The calculations where performed using copper as material for the tube, a solution with stainless steel was also computed and the results will be discussed later.

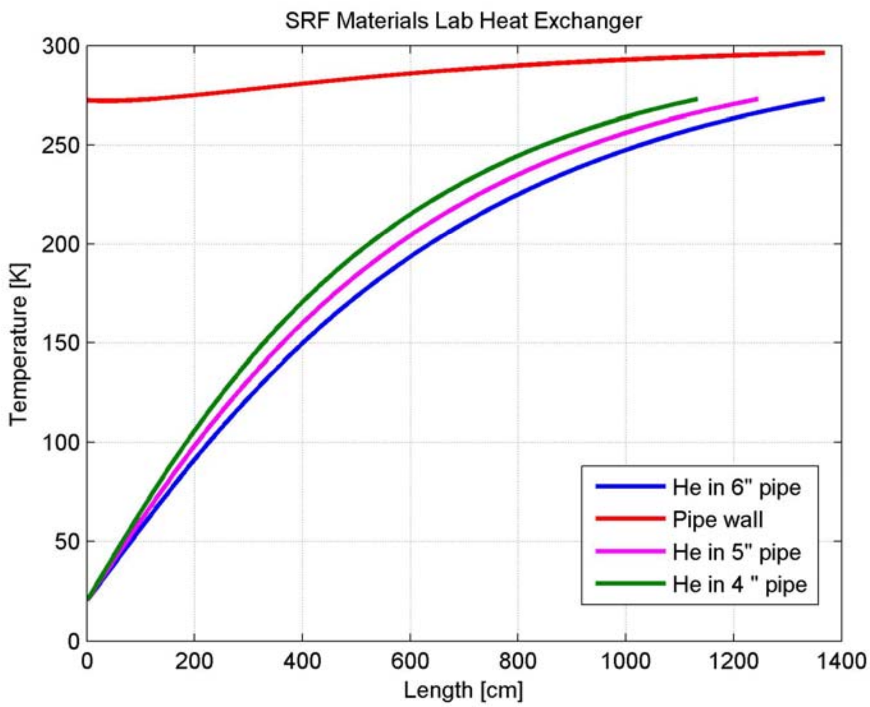

Figure 3: Temperature profile and heat exchanger length as a function of the tube diameter;

Figure 3 clearly shows that the temperature gradient is mostly present in the helium convective heat exchange with the pipe wall due to the poor film coefficient. A suitable length of the heat exchanger is in the range between 11 and 14 meters.

ANSYS SOLUTION

Ansys was used to include the component of heat conduction along the pipe neglected in the pervious calculations. A simple one dimensional model was implemented according to the scheme shown in Figure 4. The elements used for the model were:

- FLUID116 to model the helium flow mass transport, conduction and convection in the gas (using 2 auxiliary nodes)
- LINK32 to model the conduction along the pipe
- LINK34 to model the convection between air and the pipe

The helium properties were evaluated at the local temperature using a table imported by HEPAK. The pipe material properties were considered constant in the working temperature range. The air temperature was fixed at 300 K with a constant film coefficient of 20 W/m^{2}K.

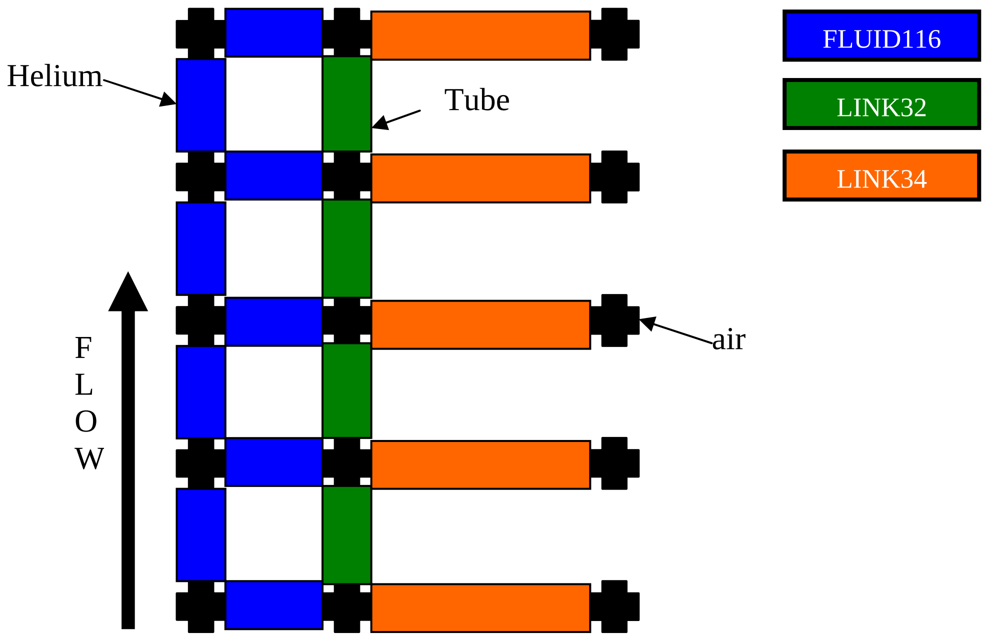

Figure 4: Schematic of the Ansys model;

The results of the calculation are shown in Figure 5. As expected the overall length of the heat exchanger is smaller with respect to the values computed using the MatLab model since the conduction effect along the pipe helps significantly the heat exchange.

In Figure 6 one can see that a suitable length of the pipe ranges between 11 and 13.5 meters depending on the pipe diameter. In order to minimize the pressure losses, the 6" tubing is recommended. The calculation was performed using two different materials for the pipe: copper with a conduction coefficient of ~307 W/mK and stainless steel with a conduction coefficient of ~16 W/mK in the considered temperature range. Figure 6 shows that using copper allows to have a little margin in the tentative of keeping the pipe temperature above 0 C to avoid frosting. The main reason for avoiding frost is its negative effect on the heat exchange coefficient. The heat exchanger pipe should be at least 14 meters long when using a diameter of 6". The pipe should be folded in a coil with 6 parallel horizontal parts 2.5 m long as shown in Figure 7. A bypass will allow reducing the pressure loss in case of high temperature gas flow.

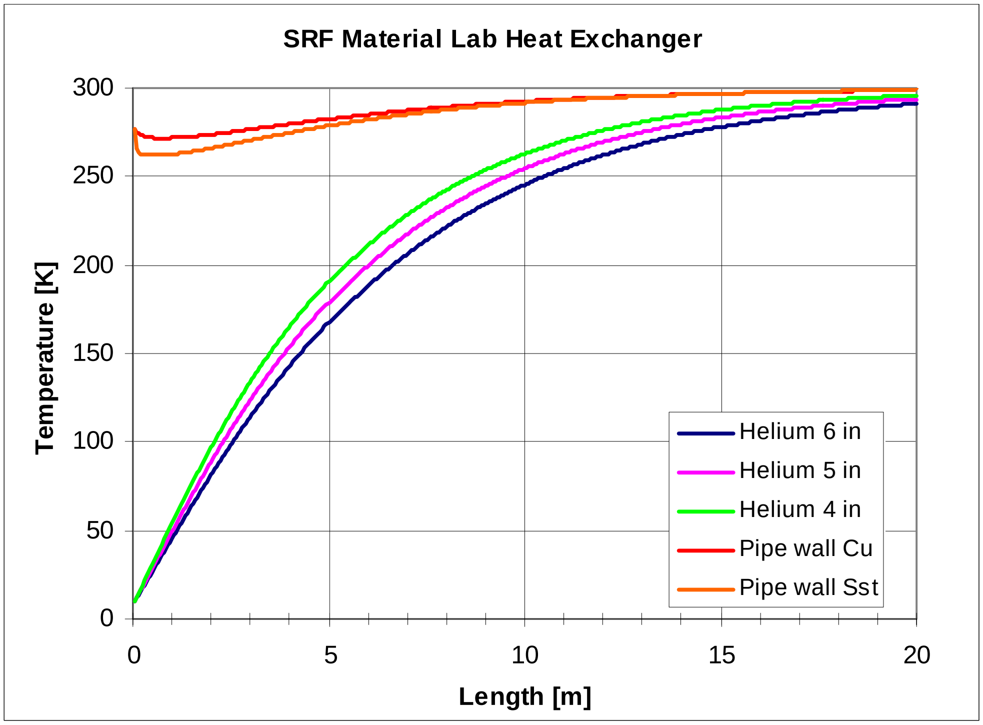

Figure 5: Temperature profile and heat exchanger length as a function of the tube diameter;

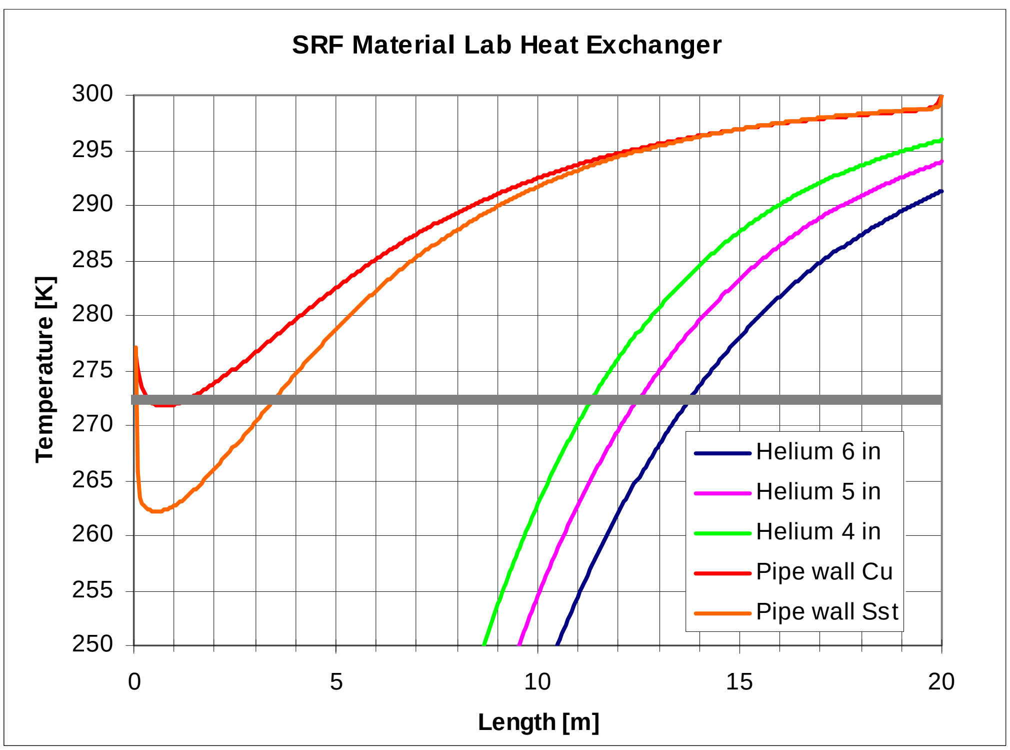

Figure 6: Temperature profile and heat exchanger length as a function of the tube diameter, high temperature zoom;

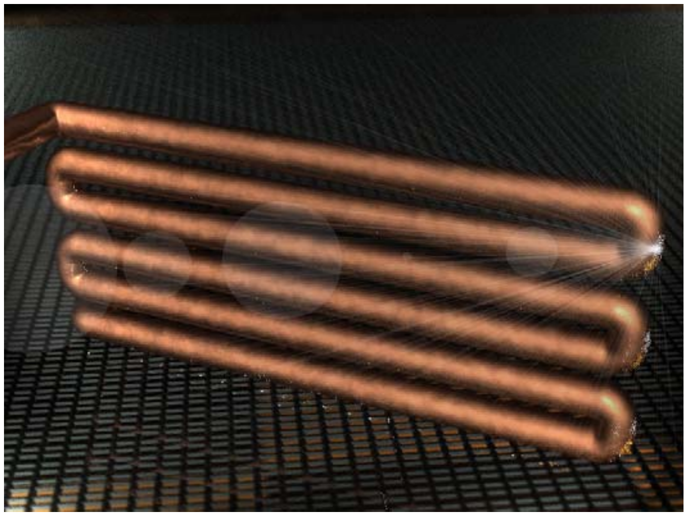

Figure 7: Heat exchanger graphical representation;

ALTERNATIVE SOLUTION

Investigations on the fabrication costs of a 6″ copper heat exchanger showed that this is a very expensive and technologically challenging solution. For cost reduction, the pipe diameter should be kept below 3″. Using a 3″ pipe would reduce the length of the heat exchanger to 10 meters but the helium speed at the high temperature side would be unacceptable (>100m/s). The alternative solution is to split the gas flow in three parallel 3″ pipes. In this case the maximum speed in the pipe would be maintained in an acceptable range below 35 m/s and the length of each pipe would be 7 meters. Figure 8 shows the temperature profile for this alternative solution.

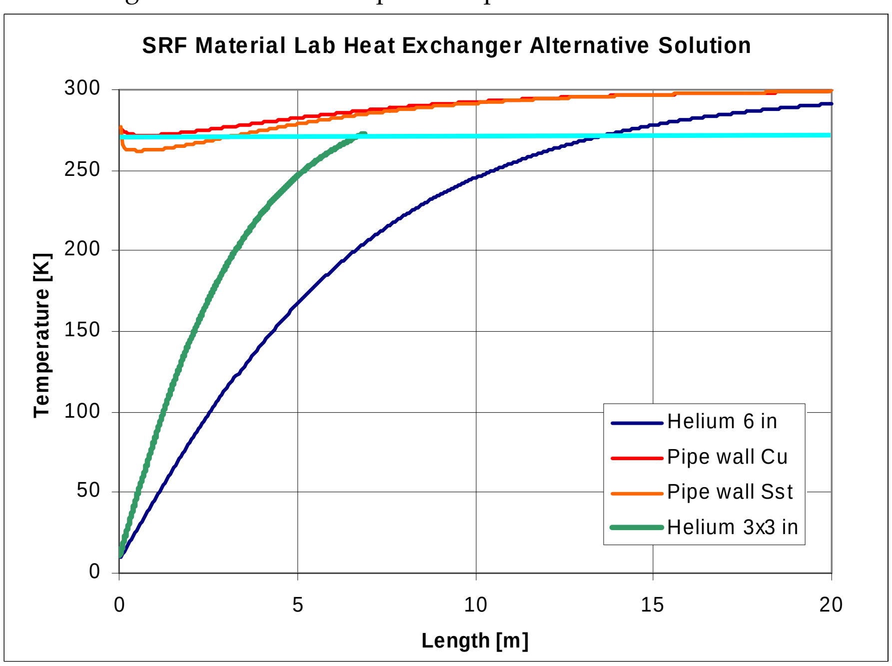

Figure 8: Temperature profile and heat exchanger length as a function of the tube diameter. Comparison between single 6″ pipe and triple 3″ pipes solutions.

CONCLUSIONS

Being the fabrication costs of a 6" heat exchanger prohibitive, the triple 3" pipes solution was chosen for the pumping system of the IB3 SRF Materials Laboratory. A model of the implementation of the solution is shown in Figure 9.

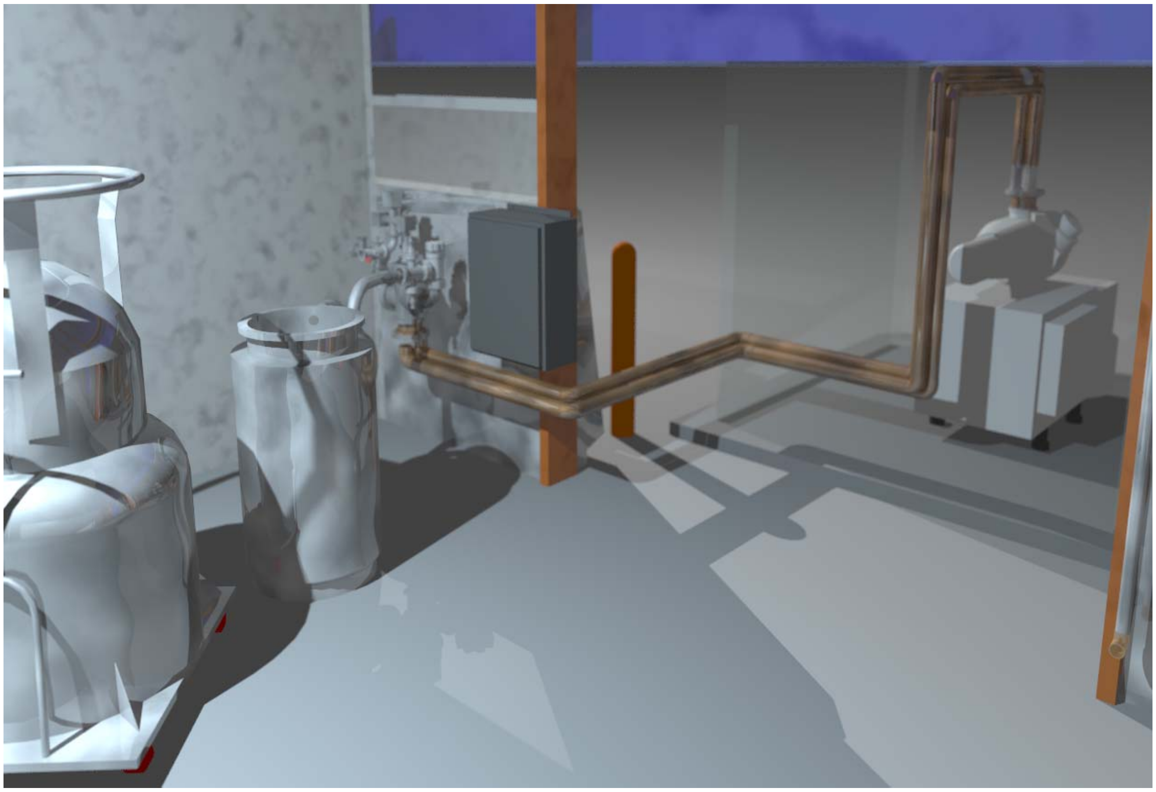

Figure 9: Solid model of the chosen solution for the pumping system heat exchanger.

PRESSURE VESSEL ENGINERING NOTE
PER CHAPTER 5031

Prepared by: Cristian Boffo_________
Preparation date: October 21, 2005 __

1. Description and Identification
Fill in the label information below:

This vessel conforms to Fermilab ES&H Manual Chapter 5031	
Vessel Title SRF Materials Lab Dewar Helium vessel – exempt from FESHM5031 LN2 shield – not exempt from FESHM5031	
Vessel Number IND-097_________________________	←**Obtain from Division/Section Safety Officer**
Vessel Drawing Number Precision Cryogenics 081904	
Maximum Allowable Working Pressures (MAWP): Internal Pressure _______19.4 PSID External Pressure _______--	
Working Temperature Range He vessel: -452°F 70 °F LN2 shield: -316 °F 70 °F	
Contents Liquid Helium, Liquid Nitrogen	
Designer/Manufacturer Precision Cryogenics Inc.	
Test Pressure (if tested at Fermi) Acceptance Date:_______	←**Document per Chapter 5034 of the Fermilab ES&H Manual**
_______PSI**G**, Hydraulic______ Pneumatic __________ Accepted as conforming to standard by ____	
of Division/Section_______________ Date:________	←**Actual signature required**

NOTE: Any subsequent changes in contents, pressures, temperatures, valving, etc., which affect the safety of this vessel shall require another review.

Reviewed by:_____________________________________ Date:____________________

Director's signature (or designee) if the vessel is for manned areas but doesn't conform to the requirements of the chapter.

____ Date: _____________________

Amendment No.:	Reviewed by:	Date:
_________________	_____________	_______________
_________________	______________	________________

Lab Property Number(s):____
Lab Location Code: 804____________________________ (obtain from safety officer)

Purpose of Vessel(s):SRF Material testing: RRR and Thermal conductivity measurements

Helium vessel:
Vessel Capacity/Size: 100 liters Diameter: 20 in _ Length: 43 in
Normal Operating Pressure (OP) 14.7 PSID
MAWP-OP = 0.3 PSID (MAWP is 15 psid for vessels to be exempt from FESHM5031)

Nitrogen shield:
Vessel Capacity/Size: 36 liters Diameter: 24 in Length: 34 in
Normal Operating Pressure (OP) 15 PSID
MAWP-OP = 6 PSID

List the numbers of all pertinent drawings and the location of the originals.

Drawing #	Location of Original
081904	Manufacturer

2. Design Verification

Is this vessel designed and built to meet the Code or "In-House Built" requirements?
Yes X No.

If "No" state the standard that was used _________________.
Demonstrate that design calculations of that standard have been made and that other requirements of that standard have been satisfied.
Skip to part 3 "system venting verification."

Does the vessel(s) have a U stamp? Yes_____ No _X___. If "Yes", complete section 2A; if "No", complete section 2B.

A. Staple photo of U stamp plate below.
Copy "U" label details to the side

	Copy data here:

Provide ASME design calculations in an appendix. On the sketch below, circle all applicable sections of the ASME code per Section VIII, Division I. (Only for non-coded vessels)

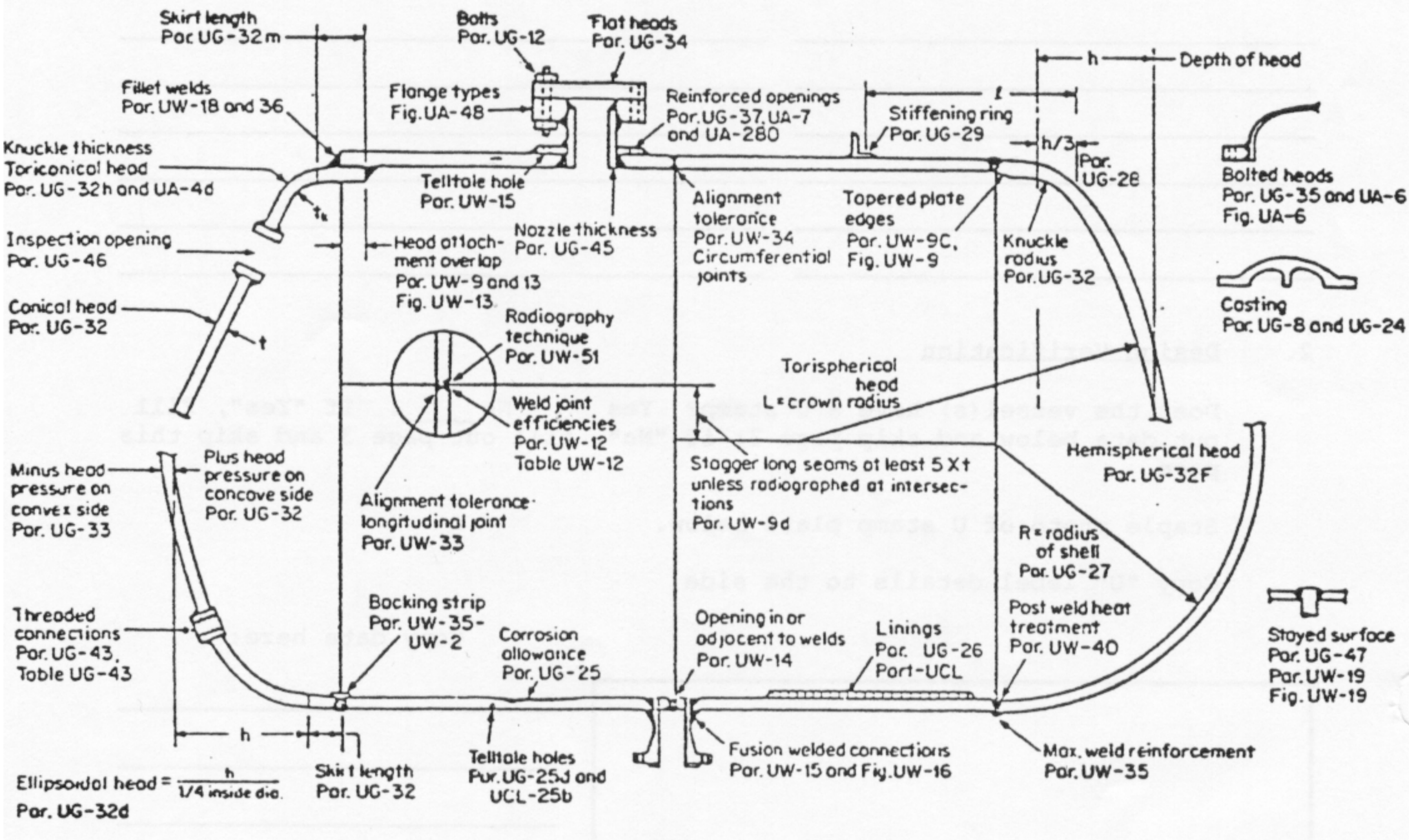

Figure 1. ASME Code: Applicable Sections

2B.

Summary of ASME Code

Item	Reference ASME Code Section	CALCULATION RESULT (Required thickness or stress level vs. actual thickness calculated stress level)
____________	____________	________ vs ________
____________	____________	________ vs ________
____________	____________	________ vs ________
____________	____________	________ vs ________
____________	____________	________ vs ________

3. System Venting Verification Provide the vent system schematic.

Does the venting system follow the Code UG-125 through UG-137?
Yes_X__ No___

Does the venting system also follow the Compressed Gas Association Standards S-1.1 and S-1.3?
Yes _X____ No_____

A "no" response to both of the two proceeding questions requires a justification and statement regarding what standards were applied to verify system venting is adequate.

List of reliefs and settings:

Manufacturer	Model #	Set Pressure	Flow Rate	Size
He: In House Parallel	Plates___	15 PSIA___	340 scfm_____	3" IPS______
N2: FNAL Parallel Plate________		15.3 PSIA_	66 scfm______	2" IPS______

4. Operating Procedure

Is an operating procedure necessary for the safe operation of this vessel?
Yes_____ No__X____ (If "Yes", it must be appended)

5. Welding Information

Has the vessel been fabricated in a non-code shop? Yes_____ No__X___
If "Yes", append a copy of the welding shop statement of welder qualification (Procedure Qualification Record, PQR) which references the Welding Procedure Specification (WPS) used to weld this vessel.

6. Existing, Used and Unmanned Area Vessels

Is this vessel or any part thereof in the above categories?
Yes_____ No__X___

If "Yes", follow the requirements for an Extended Engineering Note for Existing, Used and Unmanned Area Vessels.

7. Exceptional Vessels

Is this vessel or any part thereof in the above category?
Yes_____ No__X___

If "Yes", follow the requirements for an Extended Engineering Note for Exceptional Vessels.

THIS VESSEL CONFORMS TO FERMILAB ES&H MANUAL CHAPTER 5031

Vessel Title____

Vessel Number ____

Vessel Drawing Number ____

Maximum Allowable Working Pressures (MAWP):

Internal Pressure ___________________________

External Pressure ___________________________

Working Temperature Range ____________________________ ^{0}F _______________________ ^{0}F

Contents ____

Designer ____

Test Pressure (if tested at Fermi) DATE ___/___/_____

____ _________PSIG, Hydraulic__________________________Pneumatic ______________

Accepted as conforming to standard by

____

Of Division/Section ____

NOTE: Any subsequent changes in content, pressures, temperatures, valving, etc., which affect the safety of this vessel shall require another review and test.

Figure 2. Sample of sticker to be completed and be placed on vessel.

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ENGINEERING NOTE FOR THE IB3 SRF MATERIALS LABORATORY DEWAR

PER FESHM CHAPTER 5031

Prepared by: Cristian Boffo

Preparation Date: Oct 21, 2005

1. Description of the Dewar

This Dewar, placed in the IB3 SRF Materials Laboratory, will be used for SRF Materials testing including but not limited to: RRR, thermal conductivity, Kapitza and magnetization measurements. The Helium side of the Dewar will work in the range between atmospheric pressure (~15 PSIA) and 1 PSIA. This pressure range allows operating temperatures in liquid helium down to and below 1.5K. The Dewar will be also used as insulation and liquid helium storage for smaller inserts with the local temperature range between 1.5 K and 300 K. The Dewar will be connected to a Leybold pumping station capable of over 1100 cfm at 16 mbar and 300 K. A heat exchanger placed between the pumping station and the Dewar will assure that the helium reaches a temperature above 273 K before reaching the pumps. The Dewar drawings are provided in **appendix D**.

The Dewar itself can be divided in two separate vessels: the Helium vessel (LHe) that constitutes the main bath and the liquid nitrogen shielding (LN2). Both the LHe and the LN2 can be considered pressure vessels.

The volume of the Helium vessel is calculated as follows:

Diameter of the chamber D 20 inch

Height of the chamber H 43 inch

Volume = $(\pi \times D^2 \times H)/4 = 13{,}506\ in^3 = 7.82\ ft^3$

The volume of the Nitrogen vessel is calculated as follows:

Internal diameter D_1 21.50 inch

External diameter D_2 24.00 inch

Height of the chamber H 24.25 inch

Volume = $(\pi \times (D_2^2 - D_1^2) \times H)/4 = 2{,}166\ in^3 = 1.25\ ft^3$

The Helium vessel **does not** need to comply with FESHM 5031 since a custom designed pressure relief valve will maintain the vessel at atmospheric pressure, but mechanical calculations performed according to the ASME code are provided. The design of the parallel plates relief valve performed by Roger Rabehl is described in **appendix C** of this note.

The LN2 shielding, with an internal diameter of 21.5", an external one of 24" and a total capacity of ~35 liters, surrounds laterally the LHe vessel. The LN2 shielding **must** follow the FESHM 5031 and must be treated as an in house made pressure vessel. Calculations are provided in this note as needed.

2. Design Verification

The manufacturer performed the necessary design calculations shown in **appendix A** of this note. The calculations have been independently reviewed by Cristian Boffo following ASME Code Section VIII, Division I and are shown in **appendix B** of this note. The insulating vacuum capabilities of the vessel were tested in IB4.

3. System Venting Verification

The pressure relief devices for the Dewar have been designed according to CGA S-1.3 regulations and ASME UG-125. The devices calculations are shown in **appendix C** of this note.

4. Operating Procedures (Optional)

Does not apply.

5. Welding Information (Fermilab Shop Or Experimenter Shops)

Does not apply.

6. Extended Engineering Note for Existing Vessels, Used Vessels and Unmanned Area Vessels

Does not apply.

7. Extended Engineering Note for Exceptional Vessels

Does not apply.

APPENDIX A

PRECISION CRYOGENICS INC calculations and design of the pressure vessel.

ITEM (UG-28)

(14) OUTER CYLINDER FOR EXTERNAL PRESSURE (304 S/S)

$L = 40.6$, $D_o = 25.94$, $L/D_o = 1.565$

$t = .120$

$D_o/t = \frac{25.94}{.120} = 216.167$

$A = .000275$ (FROM Fig 5-UGO-28.0)

$B = 4,000$ (FROM Fig 5-UHA-28.1)

$P_a = \frac{4}{3}\frac{B}{D_o/t} = \frac{(4)(4,000)}{(3)(216.167)} = \frac{16,000}{648.5} = 24.67$ psi

ITEM 14 is 0.120 THK. OK ✓

---

(1) OUTER BOTTOM F&D HEAD

ITEM EXTERNAL PRESSURE (304 S/S)

(UG-33) $L_i = 26.0"$

$t_h = .187"$ (THK. of Head)

1. $\frac{L_i}{100(t_h)} = \frac{26.0}{100(.187)} = \frac{26.0}{18.70} = 1.39$

2. $\frac{L_i}{t_h} = \frac{26}{.187} = 139.04$

3. from (UHA-28.1 CHART) $B = 8,500$

4. $P_a = \frac{B}{L_i/t_h} = \frac{8,500}{26/.187} = \frac{8,500}{139.04} = 61.13$ PSI ✓

$P_a = 61.13$

.00027

ITEM (UG-27)

#13 He Vessel Shell for INTERNAL PRESSURE (304 STN. STL)

P = 60 PSIA Design Pres.

R = 10.02 inch

S = 18,700 PSI (for 304 S/S, welded)

E = .70 (NO X-RAY)

$$t(min) = \frac{PR}{SE - .6P} = \frac{(60)(10.02)}{(18,700)(.70) - (0.6)(60)} =$$

design thickness

$$= \frac{601.2}{13,090 - 36} = \frac{601.2}{13,054} = 0.046''$$ ✓ OKay

ITEM 13 is 0.120 THK.

#3 He Vessel BOTTOM F&D HEAD INTERNAL PRESSURE (304 S/S)

ITEM

(UG-32)

P = 60 PSIA Design Pressure

S = 18,700 PSI (304 S/S)

L = 20.0 (INSIDE CROWN RADIUS)

r = 1.50" (INSIDE KNUCKLE Radius)

F = 1.50 inch (Straight flg, in)

$$t(min) = \frac{0.885\ PL}{S - 0.1P} = \frac{0.885(60)(20)}{18,700 - 0.1(60)}$$

$$= \frac{1062}{18,694} = 0.057$$ ✓ OKay

ITEM 3 is 0.187 THK.

ITEM (UG-27)

(11) <u>OUTER</u>, LN2 CYL (INTERNAL PRESSURE)

ϕ 24.25 x .125 x 24.25" LG.

ALUM.

$P = 40$ psi design pressure

$R = 12.0"$

$S = 6,000$ psi (for Alum 6061 welded)

$E = .70$ (NO X-RAY)

$$t_{(min)} = \frac{PR}{SE - .6P} = \frac{(40.0)(12.0)}{(6000)(.7) - (.6)(40)} =$$

$$= \frac{480}{4,200 - 24} = \frac{480}{4176} = 0.115" \text{ Okay}$$

ITEM 11

ITEM (UG-28)

#12 INNER, LN2 CYL, EXTERNAL PRES
ALUM 6061

$L = 24.25$, $D_o = 21.50$ $L/D_o = 1.128$
$t = .125$

$$D_o/t = \frac{21.50}{.125} = 172.0$$

$A = .0006$ (FIG 5-UGO-28.0)
$B = 3,000$ (UNF-28.31)

$$P_a = \frac{4\,(3000)}{3\,(21.50/.125)} = \frac{12000}{516.00} = 23.26 \text{ psi}$$

ITEM: 12 is designed for
22 psi

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APPENDIX B

FNAL Calculations according to ASME Section VIII Div 1 and 2

1 He cylindrical vessel subjected to internal pressure

The He side of the vessel is kept at atmospheric pressure by a parallel plates relief valve, but calculations are provided. The following calculations are performed according UG-28 of ASME sec VIII Div 1.

If the thickness *t* of the cylinder does not exceed 0.356 times its radius *R* and the pressure does not exceed 0.665*S*E (where S is the maximum allowable tensile stress and E is the joint efficiency) one can use the following relationship to calculate the maximum internal pressure P to resist a circumferential stress on the joints:

$$P = \frac{SEt}{R + 0.6t} = \sim 147 \text{ PSI} \qquad \text{-1-}$$

and the following for longitudinal stress on the joints:

$$P = \frac{2SEt}{R - 0.4t} = \sim 299 \text{ PSI} \qquad \text{-2-}$$

Where

E is the joint efficiency defined in UW-12. Assuming a type 1 joint that was only visually inspected E = 0.7

S is the maximum allowable tensile stress defined for the material (AISI 304) in section II part D as 17000 PSI assuming a welded pipe

t is the thickness of the cylinder wall is 0.120”

2 He vessel elliptic bottom subjected to internal pressure

The elliptical bottom of the helium vessel according to section 4 appendix 1 ASME sec VIII Div 1 is designed to resist a pressure as follow:

$$P = \frac{2SEt}{D + 0.2t} = \sim 222 \text{ PSI} \qquad \text{-3-}$$

Where

D is the inside diameter of the bottom ~20″

t is the thickness of the bottom 0.187″

3 Conclusions

According to the code the helium vessel is designed to operate with an internal pressure of 147-15 = 132 PSIG including a reduction factor of 0.7 due to the visual weld inspection limitation. Furthermore the vessel is provided with a stiffening ring used to support the LN2 shield. The effect of the stiffening ring is not included in the above calculations.

4 LN2 cylindrical vessel subjected to internal pressure

The outer wall of the LN2 shield is considered a cylinder subject to internal pressure. The following calculations are performed according UG-28 of ASME sec VIII Div 1.

If the thickness *t* of the cylinder does not exceed 0.356 times its radius *R* and the pressure does not exceed 0.665*S*E (where S is the maximum allowable tensile stress and E is the joint efficiency) one can use the following relationship to calculate the maximum internal pressure P to resist a circumferential stress joints:

$$P = \frac{SEt}{R + 0.6t} = \sim 34.8 \text{ PSI} \qquad \text{-4-}$$

and the following for longitudinal stress on the joints:

$$P = \frac{2SEt}{R - 0.4t} = \sim 70.3 \text{ PSI} \qquad \text{-5-}$$

Where

E is the joint efficiency defined in UW-12. Assuming a type 1 joint that was only visually inspected E = 0.7

S is the maximum allowable tensile stress defined for the material (Al 6061) in section II part D and reduced by a multiplicative factor of 0.8 as required by FESHM5031 for an in-house built pressure vessel = 4800 PSI

t is the thickness of the cylinder wall 0.125″

The outer cylinder can sustain an internal pressure of ~34.8 PSIA.

The inner wall of the LN2 shield is considered a cylinder subject to external pressure. The following calculations are performed according UG-27 of ASME sec VIII Div 1.

Vessel thickness t	0.125″
Outer diameter D_0	21.50
D_0/t ratio	172 (>10)
Total length L	24.5″
L/D_0 ratio	1.1395

Using Fig. G of Section II, Part D one finds a Factor A equal to 0.00055 and implementing it into Fig. NFA-12 one finds a Factor B of ~ 2800. Finally one can calculate the maximum allowable external pressure while accounting for the FESHM5031 factor of 0.8 as:

$$P = \frac{(0.8)4B}{3\,{}^{D_0}\!/\!_{t}} = \sim 17.4 \qquad \text{-6-}$$

The inner cylinder can sustain an external pressure of ~17.4 PSIA

5 Conclusions

According to the code the liquid nitrogen shielding is designed to operate with an internal pressure of 17.4-14.6 = 2.8 PSIG.

The safety relief devices must be designed accordingly as reported in **Appendix C** of this note. However, the single vent line restricts the flow. The resulting pressure drop for the fire condition is about 4.6 psig, meaning the shield pressure is 19.2 psia. This corresponds to a stress multiplicative factor closer to 0.9 instead of the 0.8 specified by FESHM5031. The fire condition is considered the least likely failure scenario. The dewar is located in a lab space, isolated from the work floor and possible ignition sources such as welding and brazing operations. For this reason, allowing a factor of 0.9 rather than 0.8 seems reasonable.

The other failure scenarios can be adequately handled by the single vent line. For loss of vacuum due to an internal leak, the calculated flow rate is 29.8 g/s. The pressure drop for this flow rate is 1 psi. For loss of vacuum to air, the calculated flow rate is 64.4 g/s with a 4.4 psi pressure drop but the spoiled vacuum means that the shield would be operating at only 4.4 psid.

The additional vent plumbing will be sized to minimize the additional pressure drop, and the MAWP of the shield will be defined as 19.4 psid internal.

6 Finite elements analysis

Additional finite elements analysis of the structural elements of the Dewar are provided.

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APPENDIX C

VACUUM VESSEL ENGINEERING NOTE EXCLUSION
PER CHAPTER 5033

Prepared by: Roger Rabehl
Preparation Date: October 19, 2005

The vacuum vessel associated with Technical Division Pressure Vessel IND-097 (SRF Materials Lab Dewar) is excluded from the engineering note requirement of FESHM Chapter 5033 Vacuum Vessel Safety because its volumetric capacity is below 35 ft^3. The outer diameter of the vessel is 25.94 in. This yields a cross-section of 528.5 in^2, or 3.67 ft^2. The height of the vacuum vessel is 43 in, or 3.58 ft. The calculated volume of the vacuum vessel is therefore 13.2 ft^3.

The vacuum vessel is also excluded from the engineering note requirement of FESHM Chapter 5031 Pressure Vessels because sizing of the system relief devices will prevent the vacuum vessel from exceeding a 15 psig internal pressure. The parallel plate relief devices prevent the helium vessel and the thermal shield from reaching 15 psig. The vacuum vessel pressure is therefore also limited to less than 15 psig because the vacuum vessel would be in communication with the helium vessel or the thermal shield in the event of a leak.

IB3 RF Materials Lab Test Dewar: Parallel Plate Relief Sizing and Design

Roger Rabehl
September 13, 2005

A parallel plate relief device has been designed to prevent the IB3 RF Materials Lab test dewar pressure from exceeding 15 psia under any circumstances. The vessel is then exempt from the ASME Boiler and Pressure Vessel code requirements and the Fermilab ES&H Chapter 5031 Engineering Note.

This document describes the sizing and design of this parallel plate relief device.

Sizing

The first step is to determine the maximum flow rate that must be vented in order to prevent the test dewar pressure from exceeding 15 psia. Three conditions are considered: loss of insulating vacuum to helium, loss of insulating vacuum to atmosphere, and fire. The CGA S-1.3 code ("Pressure Relief Device Standards – Part 3 – Stationary Storage Containers for Compressed Gases") is used to calculate venting rates for fire and loss of insulating vacuum to helium. Published experimental results are used to calculate the venting rate for the case of loss of insulating vacuum to atmosphere. All parameters are documented and results calculated in the attached spreadsheet.

The loss of insulating vacuum to helium condition is given by Equation 1 from CGA S-1.3-1995 paragraph 5.2.2. The required venting rate is 55 scfm air.

$$Q_a = \frac{590 - T}{4\left(1660 - T\right)} F\, G_i\, U\, A \tag{1}$$

The fire condition is given by Equation 2 from CGA S-1.3-1995 paragraph 5.3.3. The required venting rate is 340 scfm air.

$$Q_a = F\, G_i\, U\, A^{0.82} \tag{2}$$

For loss of insulating vacuum to atmosphere, a heat load of 0.6 W/cm^2 of superinsulated vessel surface area is used (Lehmann and Zahn, "Safety Aspects for LHe Cryostats and LHe Transport Containers", ICEC7, London, 1978). The required venting rate is 262 scfm air (0.51 kg/s He).

These calculations indicate that the parallel plate relief device must be sized for a 340 scfm air (0.66 kg/s He) maximum venting rate at 15 psia.

Design

A sketch of the parallel plate is shown in Figure 1. The device consists of a stationary plate welded to the vent pipe, a lifting plate, a shroud, and four shoulder screws. No springs are used to push down on the lifting plate and seal it on the o-rings. The weight of the lifting plate itself accomplishes this. The four shoulder screws merely guide the lifting plate.

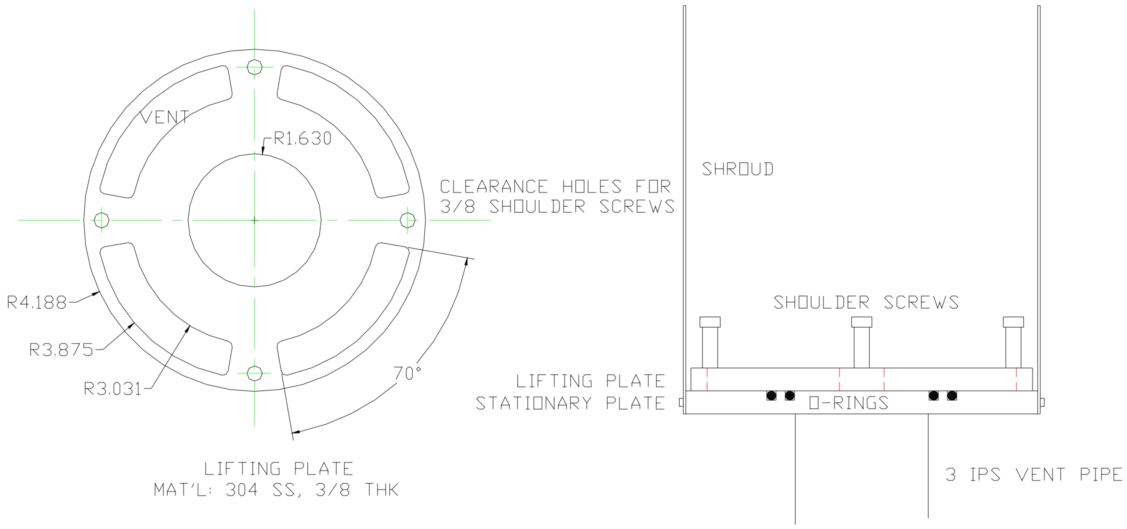


Figure 1. Lifting plate detail and parallel plate relief assembly.

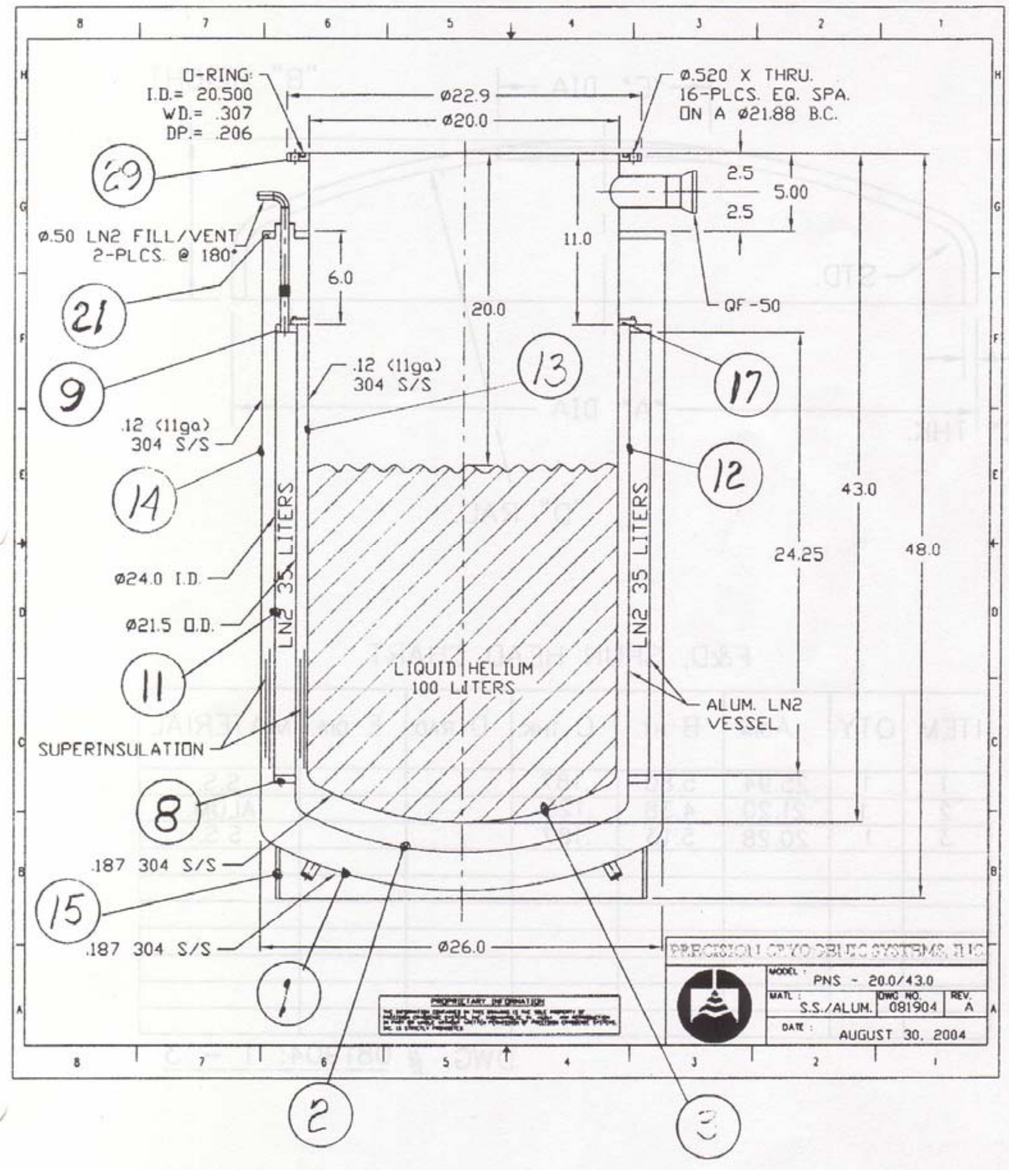
The stationary plate uses two o-rings to seal against the lifting plate and minimize leakage. There are also four threaded holes for the shoulder screws.

The lifting plate was designed so that it begins lifting at just under 0.3 psid. The four vents in the lifting plate have a larger flow area than the 3 Sch 10 vent pipe and prevent the dewar pressure from rising over 15 psia under full flow conditions. Once the shroud is in place, the flow is safely directed upward rather than outward.

The attached EES program shows the calculated ΔP when the plate begins lifting and under full flow conditions. The plate will begin lifting at 0.29 psid (14.99 psia in the test dewar), and at full flow conditions the pressure drop between the test dewar and atmosphere will be 0.22 psi (14.92 psia in the test dewar). These calculations assume a 4.375 in diameter o-ring and a 3/8 in thick stainless steel lifting plate. The plate thickness will likely be modified based on the actual o-ring diameter in the final design.

<u>Conclusion</u>

The designed parallel plate relief device will prevent the IB3 RF materials test dewar pressure from exceeding 15 psia under all conditions.


O-RING:
I.D.= 20.500
W.D.= .307
DP.= .206
ø22.9
ø20.0
ø.520 X THRU.
16-PLCS. EQ. SPA.
ON A ø21.88 B.C.
29
2.5
5.00
2.5
ø.50 LN2 FILL/VENT
2-PLCS. @ 180°
11.0
6.0
21
20.0
QF-50
9
.12 (11ga)
304 S/S
13
17
.12 (11ga)
304 S/S
14
12
43.0
LN2 35 LITERS
LN2 35 LITERS
ø24.0 I.D.
24.25
48.0
ø21.5 O.D.
11
LIQUID HELIUM
100 LITERS
ALUM. LN2
VESSEL
SUPERINSULATION
8
.187 304 S/S
15
.187 304 S/S
ø26.0
1
PROPRIETARY INFORMATION
PRECISION CRYOGENIC SYSTEMS, INC.
MODEL: PNS - 20.0/43.0
MATL: S.S./ALUM.
DWG NO. 081904
REV. A
DATE: AUGUST 30, 2004
2
3

RJR
9/8/2005

CGA S-1.3-1995 Primary Relief Valve Sizing

$$\boxed{Q_a = \frac{(590 - T)}{4(1660 - T)} F\, G_i\, U\, A} \quad (5.2.2)$$

Qa	Required flow capacity (scfm)
T	Sizing temperature (for subcritical fluids, the saturation temperature at the relieving pressure) (R)
F	Correction factor
Gi	Gas factor
U	Overall heat transfer coefficient of saturated insulating material (Btu/hr-ft^2-F)
A	Average of the inner and outer surface areas of the container insulation (ft^2)

Values

P	15	psia; relieving pressure
T	4.24	K
	7.662	R
F	1	
Gi	52.5	for helium (Table 1)
k_cond	0.037	Btu/hr-ft-F; thermal conductivity of 80 K/1 atm GHe
L	0.063	ft; conduction distance between 80 K shield and 4.5 K vessel
U	0.592	Btu/hr-ft^2-F; k_cond/L
A_LN2	21.3	ft^2; surface area of LN2 shield
A_LHe	18.8	ft^2; surface area of LHe vessel
A	20.05	ft^2; mean surface area
Qa	**55**	**scfm air**

CGA S-1.3-1995 Fire Relief Valve Sizing

$$\boxed{Q_a = F\, G_i\, U\, A^{0.82}} \quad (5.3.3)$$

Values

F	1	(from Primary Relief Sizing calculation)
Gi	52.5	(from Primary Relief Sizing calculation)
k_cond	0.122	Btu/hr-ft-F; mean thermal conductivity of 1 atm GHe between 4 K and 922 K (1200 F)
L	0.250	ft; conduction distance between 300 K vessel and 4.5 K vessel
U	0.488	Btu/hr-ft^2-F; k_cond/L
A_vac	28.1	ft^2; surface area of vacuum vessel
A_LHe	18.8	ft^2; surface area of LHe vessel
A	23.45	ft^2; mean surface area
Qa	**340**	**scfm air**

Loss of Insulating Vacuum Relief

$$\boxed{W = C\, K\, A\, P \sqrt{\frac{M}{Z\, T}}}$$

Modified from ASME Sec. VIII, Div. 1, App. 11

A_LHe	18.8	ft^2; surface area of LHe vessel
q``	0.6	W/cm^2; heat flux (Lehmann and Zahn)
q	10479	W
h_fg	20.6	J/g
m_dot	0.51	kg/s He
For helium		
W_He	4038	lbm/hr; He mass flow rate
C	378	constant for He
P	15	psia; relieving pressure
M	4	molecular weight of He
T	9	R; venting temperature
Z	0.81	compressibility factor
KA	0.961	in2; product of coefficient of discharge and discharge area
For air		
C	356	constant for air
P	15	psia; relieving pressure
M	29	molecular weight of air
T	530	R; venting temperature
KA	0.961	in2; product of coefficient of discharge and discharge area
W_air	1201	lbm/hr; air mass flow rate
rho_std_air	1.225	kg/m3; STP density of air
rho_std_air	0.0765	lbm/ft3; STP density of air
V_dot_air	**262**	**scfm air**

CALCULATE PRESSURE REQUIRED TO BEGIN LIFTING

Plate geometry

$ID = 3.26$ *in*

$OD_{plate} = 8.375$ *in*

$OD_{slot} = 7.75$ *in*

$ID_{slot} = 6.0625$ *in*

$\theta_{slot} = 280$ *degrees*

$t1 = 0.375$ *in; lifting plate thickness*

$d_{hole} = 0.375$ *in; bolt hole diameter*

$n_{hole} = 4$ *number of bolts*

$D_{oring} = 4.375$ *in*

$\rho = 7900 \cdot \left[0.00003612729 \cdot \frac{lbm/in3}{kg/m3} \right]$ *lbm/in^3; density of stainless steel*

$V = \left[\frac{\pi}{4} \cdot OD_{plate}^2 - \frac{\pi}{4} \cdot \left(OD_{slot}^2 - ID_{slot}^2 \right) \cdot \frac{\theta_{slot}}{360} - n_{hole} \cdot \frac{\pi}{4} \cdot d_{hole}^2 \right] \cdot t1$ *in^3; volume of metal*

$m = \rho \cdot V$ *lbm; mass of lifting plate*

$\Delta P_1 = \frac{m}{\frac{\pi}{4} \cdot D_{oring}^2}$ *psi; pressure differential required to begin lifting*

CALCULATE PRESSURE DROP DUE TO FLOW WHEN PLATE HAS LIFTED

$\dot{m}_{He} = 0.66$ *kg/s*

$T_{He} = 5$ *K*

$P_{He} = 14.8 \cdot \left[6894.758 \cdot \frac{Pa}{psi} \right]$ *Pa*

Call **HEPROP**$[\text{''}, 0, P_{He}, T_{He} : \rho_{He}\ x_{He}]$

Call **HEPROP**$[\text{''}, 16, \rho_{He}\ T_{He} : \mu_{He}]$

$ID_m = ID \cdot \left[0.0254 \cdot \frac{m}{in} \right]$ *m; pipe inner diameter*

$A_{flow} = \frac{\pi}{4} \cdot ID_m^2$ *m2; pipe flow area*

$Re = \frac{\dot{m}_{He} \cdot ID_m}{\mu_{He} \cdot A_{flow}}$ *Reynolds number*

$f = \textbf{Getf}[Re]$ *friction factor*

$n_{elbow} = 0$ *number of elbows*

$K_{elbow} = 30$ *elbow loss coefficient*

$K_e = 0.7 + 1$ *expansion loss coefficient*

$K_c = 0.5 + 0.2$ *contraction loss coefficient*

$L_{pipe} = 0.5 \cdot \left[0.3048 \cdot \frac{m}{ft}\right]$ *m*

$$\Delta P_2 = \left[f \cdot \left(\frac{L_{pipe}}{ID_m} + n_{elbow} \cdot K_{elbow}\right) + K_c + K_e\right] \cdot \frac{\dot{m}_{He}^2}{2 \cdot \rho_{He} \cdot A_{flow}^2} \cdot \left[0.0001450377 \cdot \frac{psi}{Pa}\right]$$

psi; pressure drop between dewar and atm when plate has lifted

$A_{flow} = 0.005385$ [m^2]
$ID = 3.26$ [in]
$K_e = 1.7$
$\mu_{He} = 0.000001392$ [kg/m-s]
$OD_{plate} = 8.375$ [in]
$\rho = 0.2854$ [lbm/in^3]
$T_{He} = 5$ [K]

$d_{hole} = 0.375$ [in]
$ID_m = 0.0828$ [m]
$K_{elbow} = 30$
$\dot{m}_{He} = 0.66$ [kg/s]
$OD_{slot} = 7.75$ [in]
$\rho_{He} = 12.09$ [kg/m^3]
$V = 15.15$ [in^3]

$D_{oring} = 4.375$ [in]
$ID_{slot} = 6.063$ [in]
$L_{pipe} = 0.1524$ [m]
$n_{elbow} = 0$
$P_{He} = 102042$ [Pa]
$t1 = 0.375$ [in]
$x_{He} = 2$

$f = 0.007803$
$K_c = 0.7$
$m = 4.325$ [lbm]
$n_{hole} = 4$
$Re = 7.289E+06$
$\theta_{slot} = 280$ [deg]

Arrays Table

	ΔP_i [psi]
1	0.2877
2	0.2176

IB3 RF Materials Lab Test Dewar: N2 Vent Line and Parallel Plate Relief Sizing

Roger Rabehl
October 19, 2005

Analysis of the LN2 shield indicates that its MAWP is 4.8 psig. The shield must be treated as a pressure vessel; however; ASME-coded relief valves cannot be purchased with a setpoint below 15 psig. The shield will therefore be protected with a Fermilab-style parallel plate relief. This parallel plate relief will also act as a check valve to prevent cryopumping during non-operational periods.

This document verifies that the Fermilab parallel plate relief has sufficient flow capacity. A preliminary relief line sizing to minimize additional pressure drop is also presented.

Venting Verification

Relief device sizing using the CGA S-1.3 code ("Pressure Relief Device Standards – Part 3 – Stationary Storage Containers for Compressed Gases") indicates that the fire condition is the limiting condition. A GN2 flow rate of 71 g/s is required.

The pressure drop in the dewar shield vent tubing has been previously calculated to be 4.6 psi. This leaves a maximum pressure drop of about 0.2 psi for the vent line and the parallel plate relief.

The vent line pressure drop is calculated by Equation 1:

$$\Delta P = f\left(\frac{L}{D}\right)\left(\frac{\dot{m}^2}{2\,\rho\,A^2}\right) \qquad (1)$$

where ΔP is the pressure drop, f is the friction factor, L is the line length (assumed to be 10 ft), D is the line diameter, m is the mass flow rate (0.071 kg/s), ρ is the density, and A is the flow area.

The minimum recommended inner diameter for a rigid vent line is 1.5 in. A 100 K vent flow results in a 0.09 psi pressure drop. To achieve similar results with a corrugated, flexible line the minimum inner diameter is 2 in due to a friction factor increase of 4x.

The parallel plate is approximated by Equation 2 as an uncontrolled expansion from a 2.625 in diameter port to atmosphere:

$$\Delta P = K\left(\frac{\dot{m}^2}{2\,\rho\,A^2}\right) \qquad (2)$$

where K is the expansion loss coefficient (= 1). The calculated pressure drop due to uncontrolled expansion is much less than 0.1 psi.

Conclusion

The recommended minimum inside diameter for the shield vent line is 1.5 in for a rigid line and 2 in for a corrugated, flexible line. The combined pressure drops between the test dewar and atmosphere will then allow the thermal shield to remain below its MAWP.

Name	Sym	Relationship	Units	Reference	
Diameter	Di		in		21.500
	Do		in		24.000
Length	l		in		24.250
Plate surface	Ap	pi*D^2/4/144	ft^2		0.620
Lateral surface	Al	pi*D*l/144	ft^2		24.067
Total surface	A	Ap+Al	ft^2		24.688
Pressure setting	P		PSI		15.000
Gj factor	Gj			CGA S-1.3 Table 1	10.200
f factor	f			CGA S-1.3	1.000
Temperature Nitrogen	T		R	according to IND-088	139.000
N2 thermal cond (max of 225 K He and 225 K N2)	k1		BTU/(hr ft F)		0.074
N2 thermal cond	k2		BTU/(hr ft F)	CGA S-1.3 Table 3	0.023
Thickness of insulation	x		in	drawing	0.600
Total heat transfer coeff	U1	k1/x*12	BTU/(hr ft^2 F)	CGA S-1.3 5.2.2 5.3.3	1.480
Total heat transfer coeff	U2	k2/x*12	BTU/(hr ft^2 F)	CGA S-1.3 5.2.2 5.3.3	0.465
CGA S-1.3 par. 5.2.2				**Relief Valve**	
Relief valve capacity	**Qa**	**(590-T)/(4*(1660-T)*f*Gj*U1*A**	**scfm**	**CGA S-1.3 5.2.2**	**27.627**
Standard air density	rho	ideal gas eq.	lb/ft^3	at 60 F and 15 PSIA	0.075
Equivalent air mass flow	Wa	Qa*rho*60	lb/hr		124.154
Constant air	Cair			App. 11 ASME Sec VIII Div 1	356.000
Molecular weight air	Mair				28.900
Standard air temperature	Tair		R		520.000
KAP	KAP	Wa/Cair*sqrt(Tair/Mair)		App. 11 ASME Sec VIII Div 1	1.479
Molecular weight Nitrogen	Mn				28.000
Constant Nitrogen	Cn			App. 11 ASME Sec VIII Div 1	356.000
Nitrogen temperature	Tn		R		139.000
Nitrogen mass flow	**Wn**	**Che*KAP*sqrt(Mhe/The)**	**lb/hr**	**App. 11 ASME Sec VIII Div 1**	**236.367**
		Whe/7.93	**g/s**		**29.808**
CGA S-1.3 par. 5.3.3 fire				**Rupture disks**	
Relief valve capacity	**Qa**	**Gj*f*U2*A^0.82**	**scfm**		**65.709**
Standard air density	rho	ideal gas eq.	lb/ft^3	at 60 F and 15 PSIA	0.075
equivalent air mass flow	Wa	Qa*rho*60	lb/hr		295.296
Constant air	Cair			App. 11 ASME Sec VIII Div 1	356.000
Molecular weight air	Mair				28.900
Standard air temperature	Tair		R		520.000
KAP	KAP	Wa/Cair*sqrt(Tair/Mair)		App. 11 ASME Sec VIII Div 1	3.519
Molecular weight Nitrogen	Mn				28.000
Constant Nitrogen	Cn			App. 11 ASME Sec VIII Div 1	356.000
Nitrogen temperature	Tn		R		139.000
Nitrogen mass flow	**Wn**	**Che*KAP*sqrt(The/Mhe)**	**lb/hr**	**App. 11 ASME Sec VIII Div 1**	**562.189**
		Whe/7.93	**g/s**		**70.898**
Vacuum to air loss				**Relief Valve**	
specific heat leak	q		W/cm^2	Lehmann and Zahn	0.600
Heat of Vaporization Nitrogen	C		J/g		198.000
Nitrogen mass flow	**Wn**	**q*A/C*10000*0.0929**	**g/s**		**69.500**
		Whe*7.93	**lb/hr**		**551.13107**
Molecular weight Nitrogen	Mn				28.000
Constant Nitrogen	Cn			App. 11 ASME Sec VIII Div 1	356.000
Nitrogen temperature	Tn		R		139.000
KAP	KAP	Whe/Che*sqrt(The/Mhe)		App. 11 ASME Sec VIII Div 1	3.449
Constant air	Cair			App. 11 ASME Sec VIII Div 1	356.000
Molecular weight air	Mair				28.900
Standard air temperature	Tair		R		520.000
Standard air density	rho	ideal gas eq.	lb/ft^3	at 60 F and 15 PSIA	0.075
Equivalent air mass flow	Wa	Cair*KAP*sqrt(Mair/Tair)	lb/hr		289.488
Relief valve capacity	**Qa**	**Wa/rho/60**	**scfm**	**CGA S-1.3 5.2.2**	**64.417**

Calculation of pressure drop in the test dewar thermal shield vent line

Vent line ID	0.428	in
	10.8712	mm
Number of vent lines	1	
Vent flow area	92.77	mm^2
	9.28E-05	m^2
Mass flow rate	0.071	kg/s

	length	**[ft]**	**length [m]**	**diameter [m]**	**speed [m/s]**	**density [kg/m3]**	**viscosity [Pa*s]**	**distributed losses [Pa]**	**elbow [number]**	**tee [number]**	**resuctions [number]**	**valves [number]**	**concentrated losses [Pa]**	**TOTAL**	
total line		0.7	0.21336	0.011	383	2.00E+00	1.20E-05	31470.0592	0	0	0	0	0	31470	**Pa**
														4.56	**PSI**

Re	6.93E+05	
f	0.0027378	
ΔP/L	147497	**Pa/m**
r lv	1.46E+05	**Pa**

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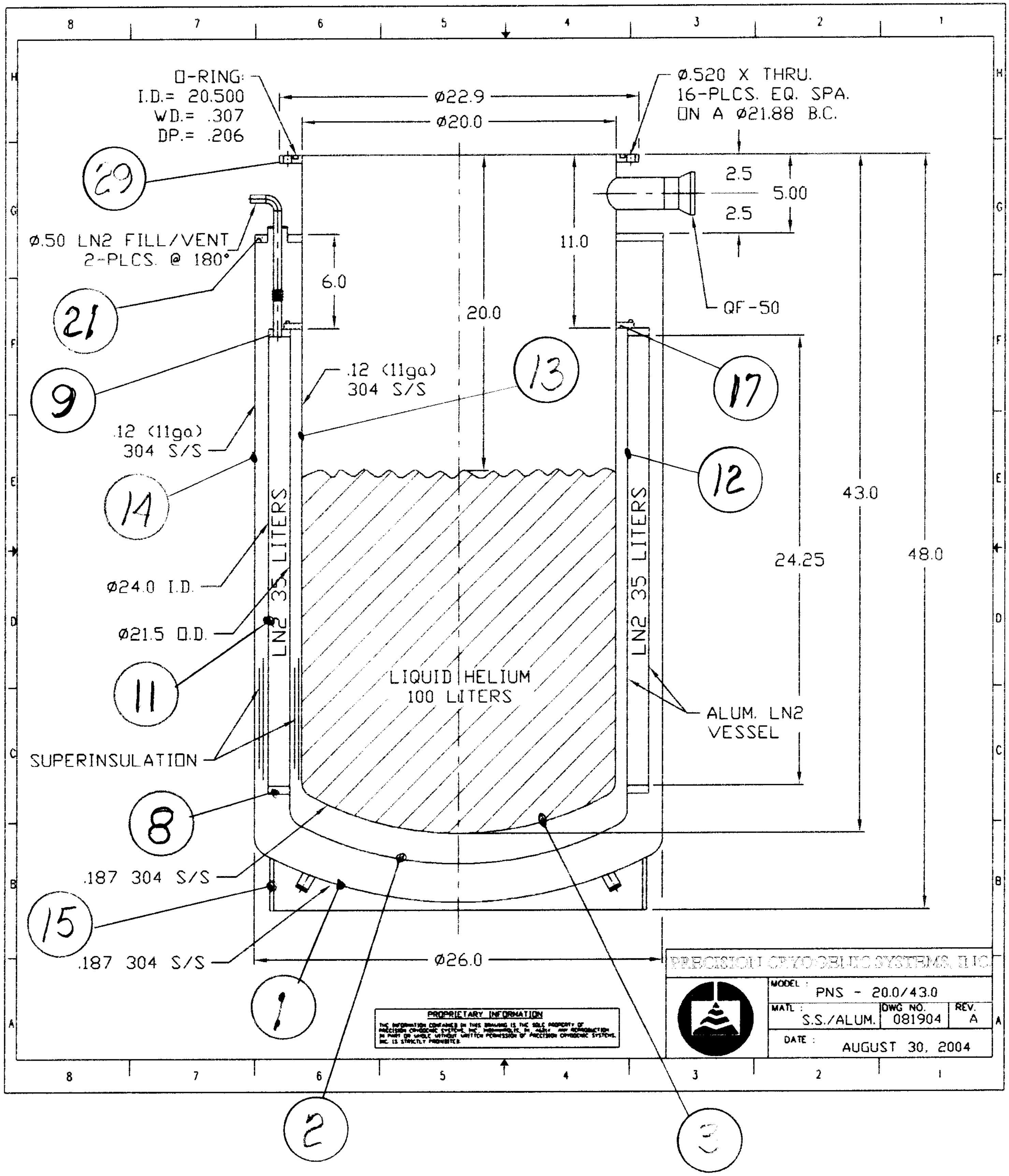
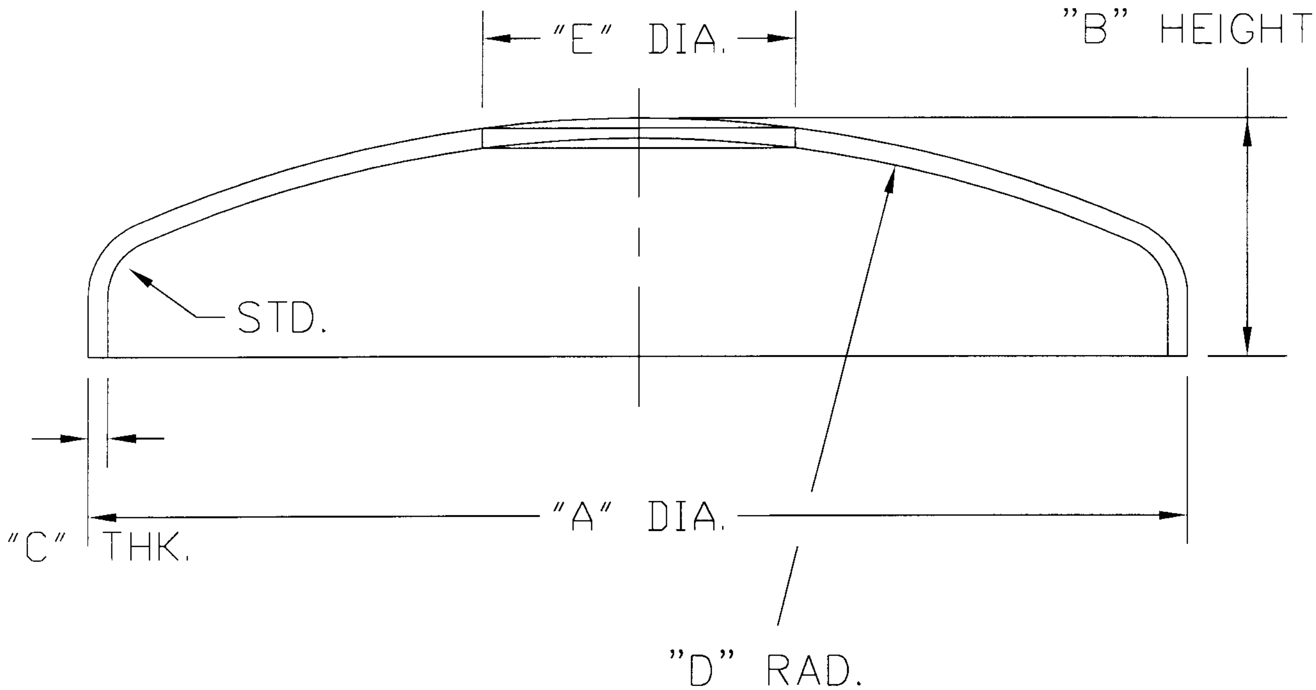
APPENDIX D

Drawings provided by the manufacturer

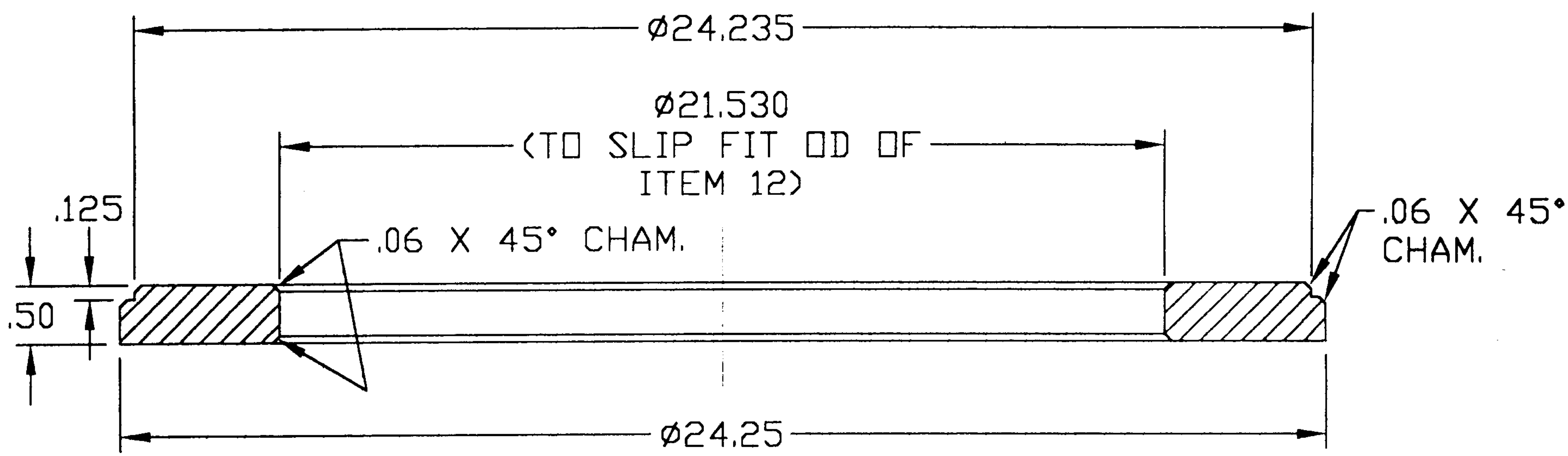

O-RING:
I.D.= 20.500
WD.= .307
DP.= .206
Ø22.9
Ø20.0
Ø.520 X THRU.
16-PLCS. EQ. SPA.
ON A Ø21.88 B.C.
2.5
5.00
2.5
Ø.50 LN2 FILL/VENT
2-PLCS. @ 180°
6.0
20.0
11.0
QF-50
.12 (11ga)
304 S/S
.12 (11ga)
304 S/S
43.0
48.0
24.25
LN2 35 LITERS
LN2 35 LITERS
Ø24.0 I.D.
Ø21.5 O.D.
LIQUID HELIUM
100 LITERS
ALUM. LN2
VESSEL
SUPERINSULATION
.187 304 S/S
.187 304 S/S
Ø26.0
29
21
9
13
17
14
12
11
8
15
1
2
3
PROPRIETARY INFORMATION
PRECISION CRYOGENIC SYSTEMS, INC.
MODEL : PNS - 20.0/43.0
MATL : S.S./ALUM.
DWG NO. 081904
REV. A
DATE : AUGUST 30, 2004



F&D, SPUN HEAD CHART

ITEM	QTY.	A DIA.	B HT.	C THK.	D RAD.	E DIA.	MATERIAL
1	1	25.94	5.80	.187			S.S.
2	1	21.20	4.38	.125			ALUM.
3	1	20.28	5.13	.187			S.S.

DWG. # 081904: 1 – 3

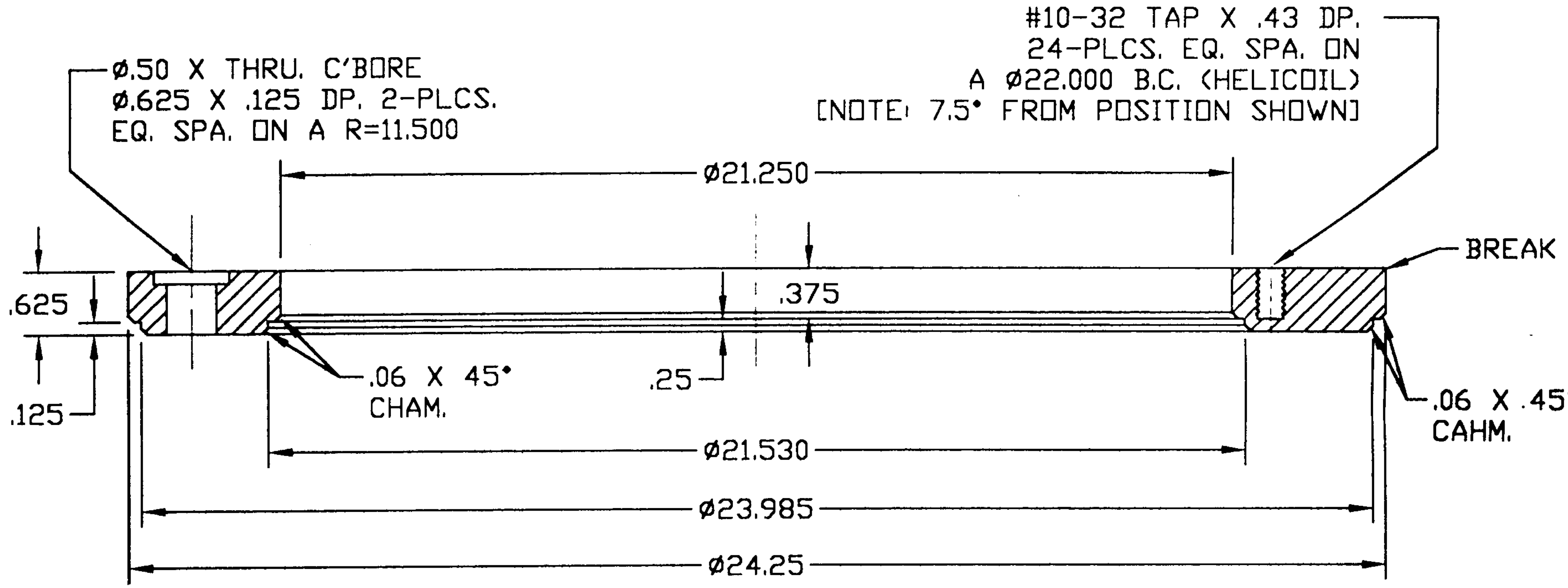


LN2 BOTTOM PLATE

MATL: ALUM.
QTY: 1-EA.

081904-8

-8

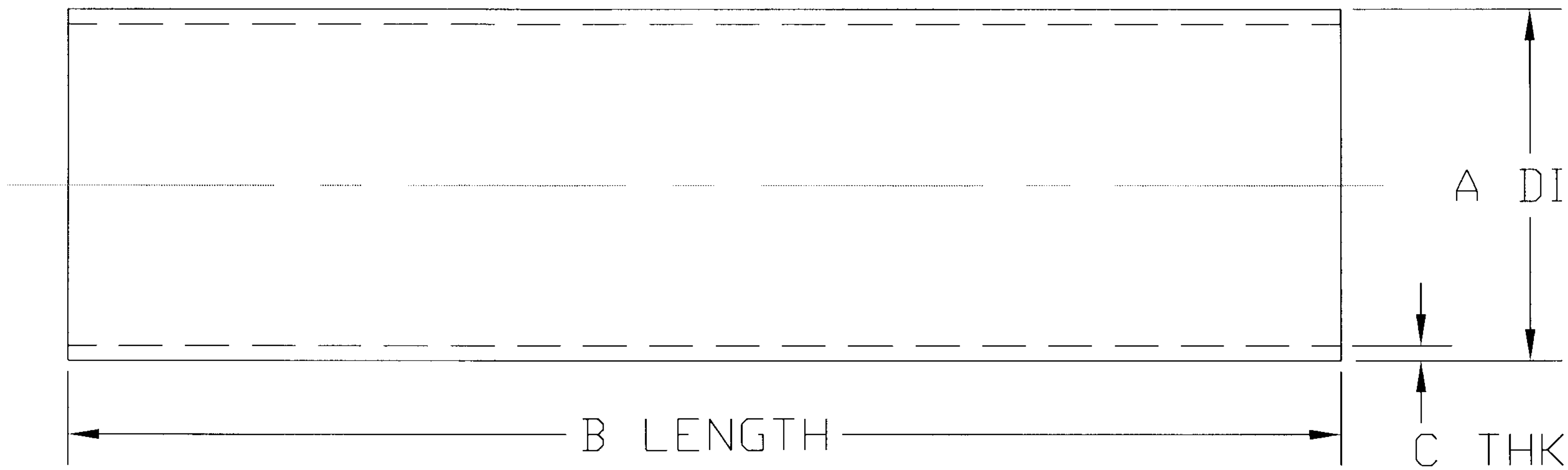


LN2 TOP PLATE

MATL: ALUM.
QTY: 1-EA.

081904-9

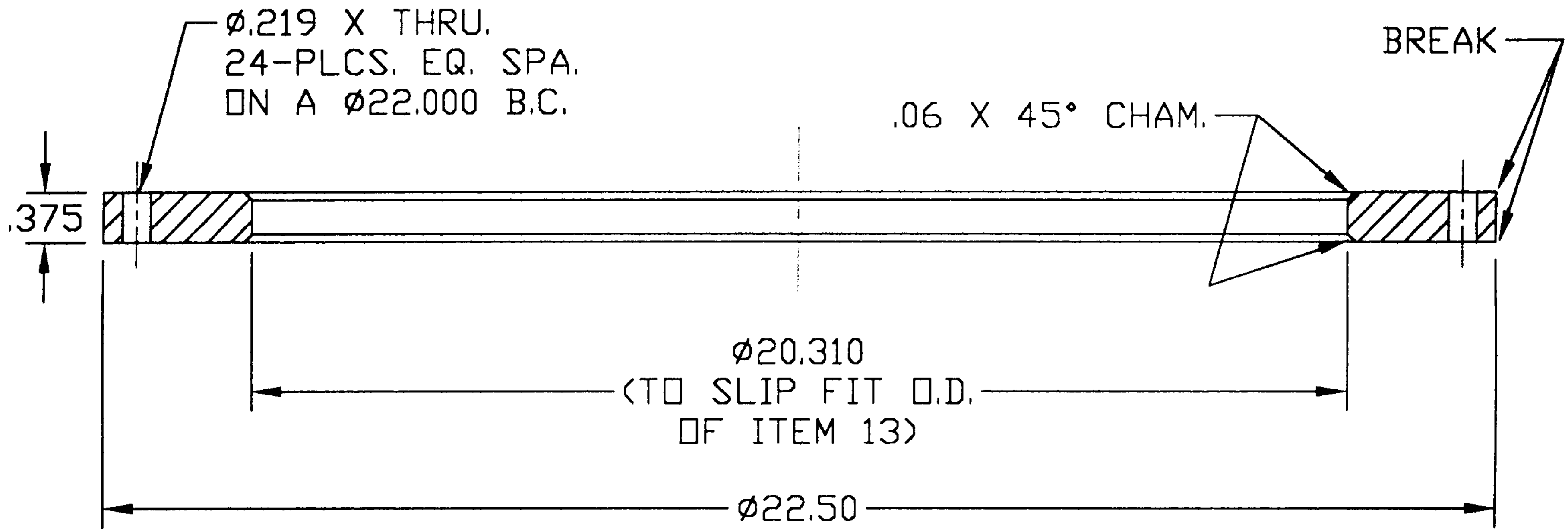
-9



CYLINDER CHART

ITEM	QTY.	A DIA.	B LGTH	C THK.		MATL:	CIRCUM:
11	1	24.25	24.25	.125		ALUM.	75.791
12	1	21.50	28.75	.125		ALUM.	67.151
13	1	20.280	37.748	.120		S.S.	63.334
14	1	25.94	35.95	.120		S.S.	81.116
15	1	24.00	4.28	.120		S.S.	75.020

DWG. # 081904: 11-15



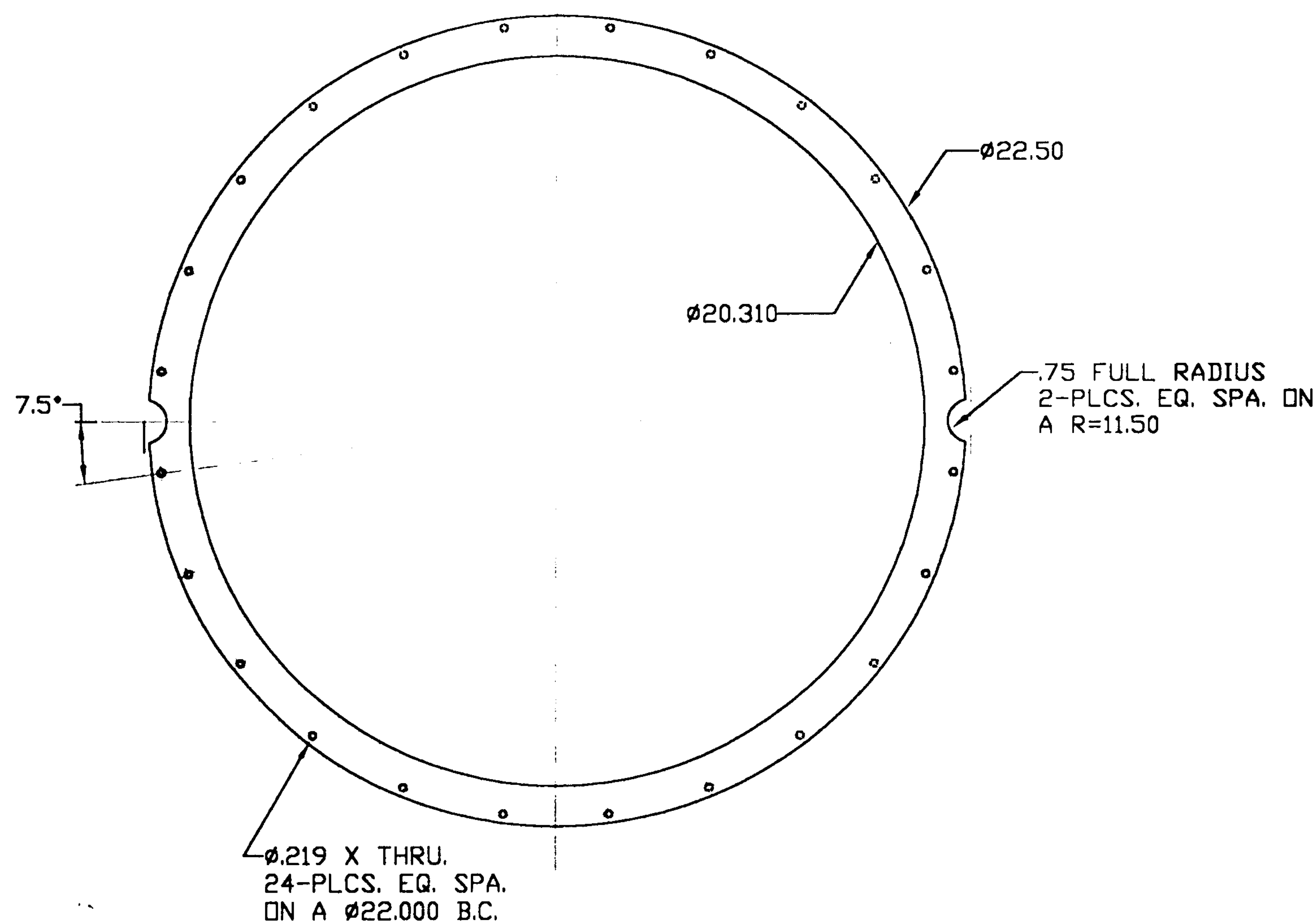
LN2 SUPPORT FLANGE

(SHEET: 1 OF 2)

MATL: S.S.
QTY: 1-EA.

081904-17

-17

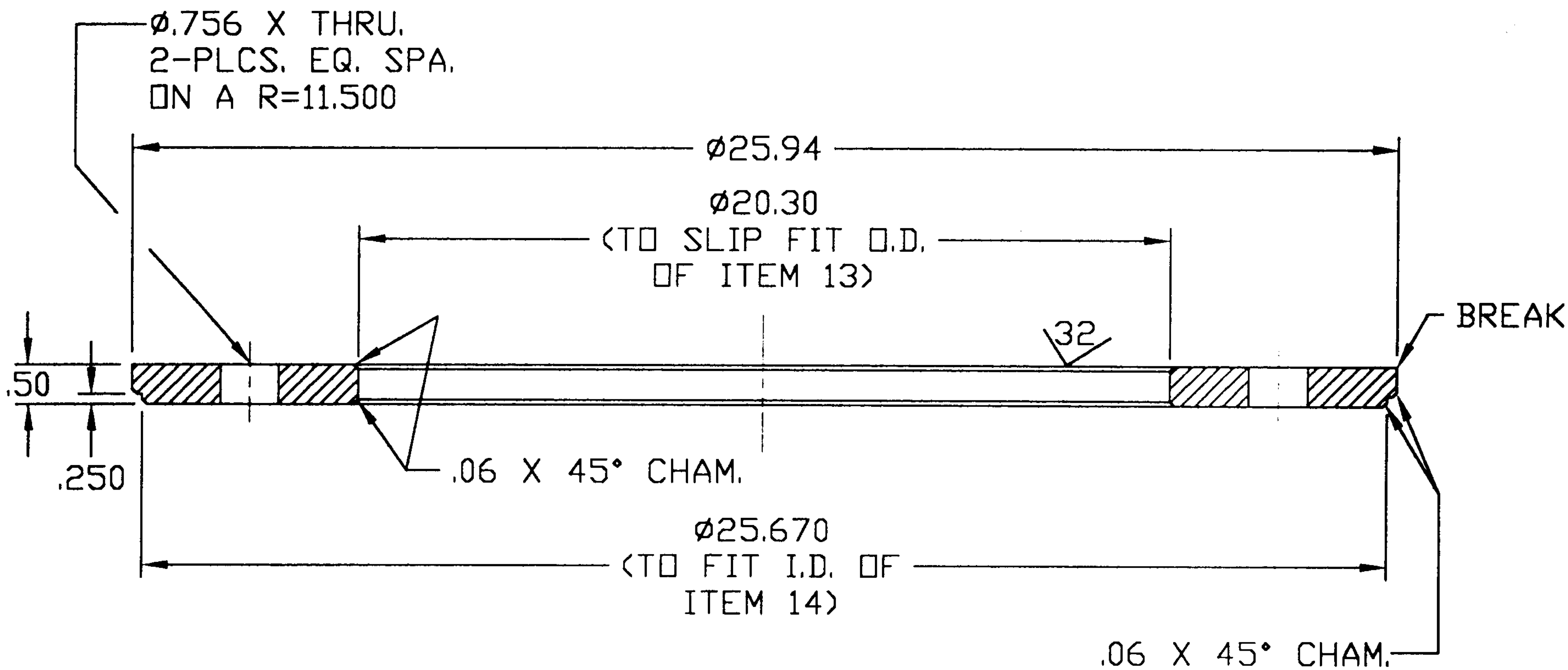


LN2 SUPPORT FLANGE

(SHEET: 2 OF 2)

MATL: S.S.
QTY: 1-EA.

081904-17

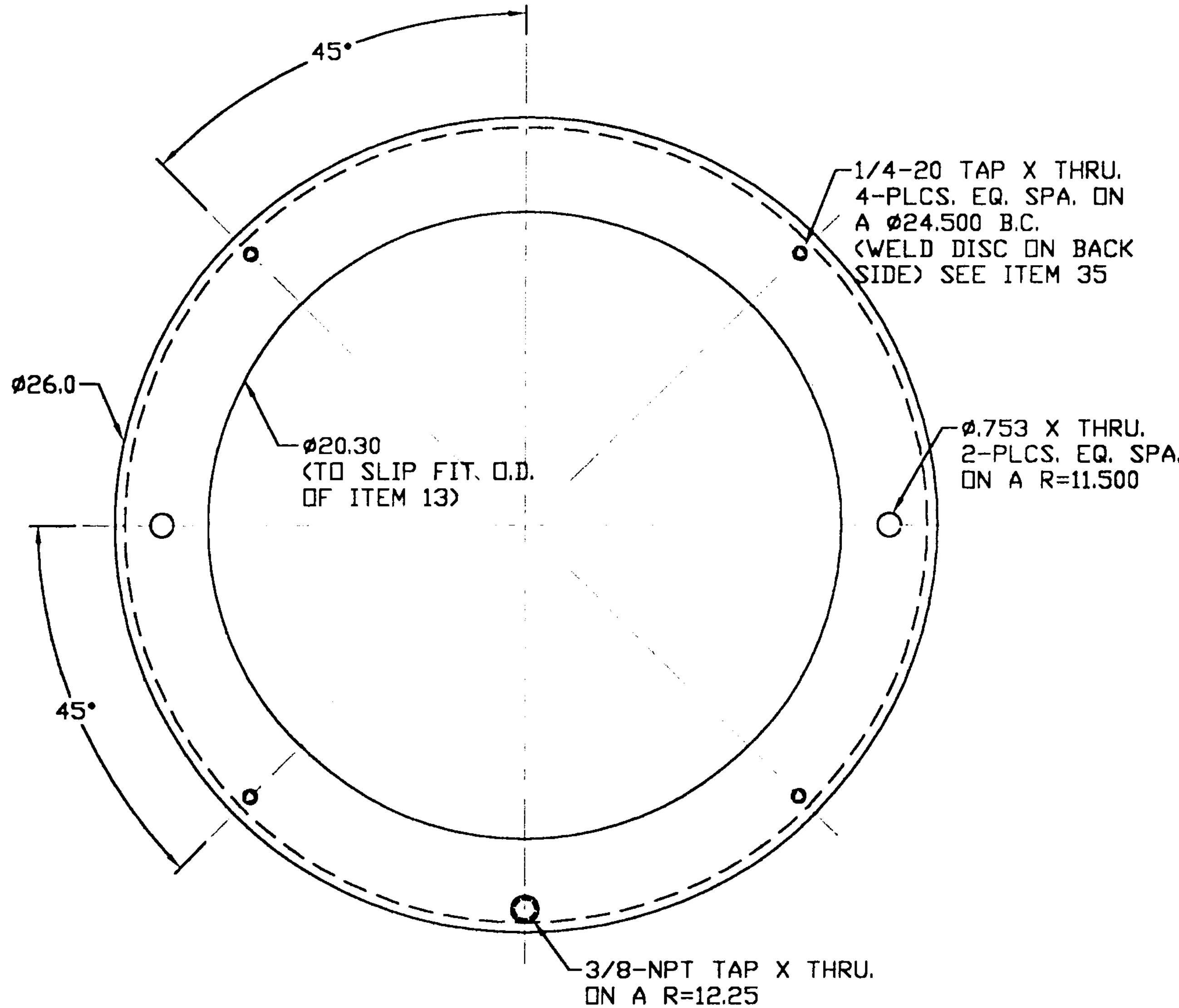


R/T FLANGE

(SHEET: 1 OF 2)

MATL: STN.-STL.
QTY: 1-EA.

081904-21



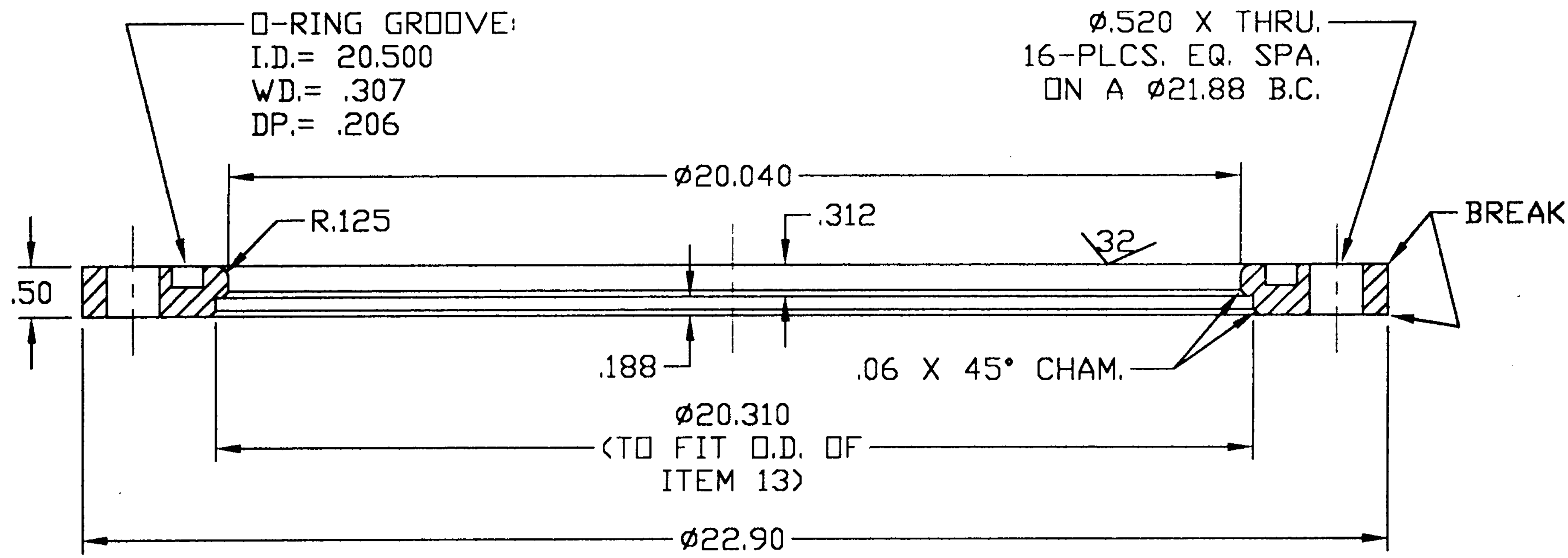
R/T FLANGE TOP VIEW

(SHEET: 2 OF 2)

MATL: STN.-STL.
QTY: 1-EA.

081904-21

12-



TOP FLANGE

MATL: S.S.
QTY: 1-EA.

081904-29